EFFECT OF OPERATING VARIABLES ON THE RECTIFICATION OF BENZENE AND TOLUENE

Approved:

Approved:

Dean of the Graduate School

EFFECT OF OPERATING VARIABLES ON THE RECTIFICATION OF BENZENE AND TOLUENE

DISSERTATION

Presented to the Faculty of the Graduate School of
The University of Texas in Partial Fulfillment
of the Requirements

For the Degree of

DOCTOR OF PHILOSOPHY

By

Paul Bennett Stewart, B. S. in Chem., M. S. in Ch. E.

Austin, Texas

June 1945

PREFACE

Distillation as an art goes back to the Middle Ages at least, as evidenced by alchemical prints[12*] showing various early forms of equipment. Particularly prominent up to the present century were destructive distillation processes, and the distillation of wines and fermented mashes to yield brandies and other spirituous liquors.

Until the appearance of treatises by Sorel[53] and by Hausbrand,[18] and indeed even to the present day, knowledge of the subject was almost entirely empirical and uncoordinated with basic scientific laws. Sorel treated the distillation of binary mixtures mathematically from the standpoint of heat and material balances, and thus laid the foundation for the science of distillation.

Since then steady and progressive applications of mathematics, physics and chemistry have been made. Current lines of work include refinements of present methods of calculation for binary mixtures, for three-component and more complex mixtures, and for design of equipment.

Peculiarly enough, although there is a very active interest

---

*Superscripts refer to references listed under "Literature Cited" at the end of the thesis.

in distillation, the great majority of the articles on the subject are theoretical in nature and do not represent new distillation data under commercial or semi-commercial conditions. In fact, no published data are available on semi-commercial bubble-tray columns to comprehensively show the effect of operating conditions, in particular, reflux ratio and point of introduction of feed. This work was undertaken to shed some light on these very important factors for bubble-cap columns.

ACKNOWLEDGEMENT

Grateful acknowledgement is due to the many people who have contributed to this work: to Professor John Griswold for direction of the project, to Robert H. Bowden for help in column construction and in the purification of the hydrocarbons, to the storeroom and shop staffs of the chemical engineering department for many favors and services cheerfully performed, and to various staff members and graduate students for helpful suggestions.

The University Research Institute supported the successful conclusion of the work by a research grant.

Austin, Texas

December, 1944

CONTENTS

LIST OF FIGURES

LIST OF TABLES

I

INTRODUCTION

There are several treatises on distillation,[22,47] and many articles pertaining to this subject are to be found in the periodical literature. But relatively little has appeared on the experimental determination of the many variables affecting operating efficiency of bubble-tray columns. Many authors[1,6,7,9,27,28,39,43,44,46] have published works purporting to show the effect of changing one or more of the operating variables, but almost without exception, these are based on theoretical considerations with no direct experimental data to verify the conclusions reached.

THEORETICAL CONSIDERATIONS

Sorel[58] and Hausbrand[18] developed rectification calculations in terms of "theoretical plates;" a theoretical plate being considered as one on which the liquid had a uniform composition and from which the vapor attained equilibrium concentration.

Later, McCabe and Thiele[30] by use of the additional approximations, partly stated and partly implicit, of; (1) equal molar latent heats of the components, (2) negligible heat of mixing, and (3) negligible sensible heat effects, noted that concentrations of vapor entering a plate were linear with concentrations of liquid on that plate for fixed operating conditions. This observation was developed into a rapid graphical method of calculating the number of theoretical plates to effect a given separa-

tion of a binary mixture.

The number of theoretical plates divided by the number of actual plates is designated "overall column (or plate) efficiency." The McCabe-Thiele method and overall plate efficiency are used universally.

Murphree[32] noted that in most instances the actual number of plates was considerably greater than the theoretical number, since the vapor was not fully enriched to its equilibrium concentration with respect to the plate liquid. Simultaneous enrichment of the vapor by stripping the liquid of its more volatile component is a rate process governed by the laws of diffusion. By applying the diffusion equations in the forms available at that time Murphree developed individual plate efficiency equations applicable to point conditions with respect to the liquid. These equations expose the major variables affecting plate efficiency, according to the diffusional mechanism. They have been reaffirmed by Griswold[16] from the more modern diffusion equations, and in modern notation are:

For controlling resistance in the vapor film,

$$\eta_{M,V} = \frac{y_n - y_{n-1}}{y_n^* - y_{n-1}} = 1 - e^{-f_V A\Theta} \qquad \ldots 1$$

For controlling resistance in the liquid film,

$$\eta_{M,L} = \frac{y_n - y_{n-1}}{y_n^* - y_{n-1}} = 1 - e^{-f_L A\theta/m} \quad \ldots \quad 2$$

where

$\eta_{M,V}$ = Murphree vapor film efficiency

$\eta_{M,L}$ = Murphree liquid film efficiency

y_{n-1} = Molar composition of vapor entering plate "n".

y_n = Molar composition of vapor leaving plate "n".

y_n^* = Molar composition of vapor in equilibrium with liquid on plate "n".

f_v = Vapor film coefficient

f_L = Liquid film coefficient

A = Interfacial vapor-liquid area

θ = Effective time of contact of liquid and vapor

m = Slope of equilibrium (y^*-x) curve at average composition of vapor between y and y_{n-1}

Equations 1 and 2 provide a theoretical basis for calculating and interpreting plate efficiencies. Numerical values are obviously the same regardless of which film is controlling. However, these equations are strictly applicable only to point conditions of liquid concentration, or to plates on which the liquid composition is constant. In large columns the liquid flow across the plate is such as to give semi-counterflow contact with the vapor rising through it from below. Lewis[28] developed mathematical relations between the Murphree or "local" plate efficiency and the "overall plate" efficiency for the

more common arrangements of vapor and liquid flow in commercial columns.

Lewis also developed relations between overal column efficiency and Murphree efficiencies for the rare case in which y^* and x are linear. The corresponding relations for the general case have not been developed, and promise to be extremely complex.

In small columns (containing only one or two bubble caps per plate) liquid concentration differences at various points on a plate are much smaller than in large commerical columns, and can be satisfactorily taken as constant at an average value for use in calculating plate efficiencies. This enables the local Murphree efficiency in the simple form of equations 1 and 2 to be applied to an entire plate in small columns.

SUMMARY OF PREVIOUS EXPERIMENTAL WORK

Design Variables

Construction of a column involves bubble cap design, plate spacing, plate arrangement, down pipe size and arrangement, and other factors. Obviously, when a column is once built, barring changes in it, these items become constant for that particular unit. Plate spacing has probably been investigated more extensively than any other design variable, particularly in regard to its effect upon entrainment. Work on this subject includes articles by Sherwood and Jenny,[50] by Holbrook and Baker,[19] and by Singer, Wilson and Brown.[51] Decrease in plate spacing increases entrainment, thereby reducing the allowable vapor velocity and decreasing separation. As might be suspected,

foaming characteristics of the liquid rather than plate spacing sometimes controls allowable vapor velocity.

Effect of Vapor Velocity

Maximum allowable vapor velocity is of major importance since it determines the capacity of a column. Brown and Lockhart[5] present data on a 30-plate gasoline fractionating column six feet in diameter and 85 feet high. They show that efficiency is higher in the stripping than in the enriching section, and that over most of the range covered, the overall efficiency is essentially independent of vapor velocity but drops off in both sections at low velocities. Peavy and Baker[41] gave results on a 3-plate, 18-inch diameter laboratory column operated on ethanol-water. They conclude that the Murphree efficiency reaches a maximum near the middle of the operable vapor velocity range, and that the higher the liquid level on the plate the higher the efficiency. Carey, Griswold, Lewis and McAdams[8] report data obtained on a 10-plate column operated on benzene-toluene, concluding that the plate efficiency is essentially independent of vapor velocity except at high and low extremes of velocities where the efficiency decreases somewhat. Kirschbaum and Andrews,[24] from rectification data on ethanol-water, conclude that efficiency reaches a maximum at some definite vapor velocity, and is also a function of reflux ratio, the higher reflux ratios giving greater efficiencies.

Effect of Liquid Concentration and Viscosity

Liquid concentration in relation to its effect on efficiency

has been studied experimentally for the ethanol-water system by Kirschbaum,[23] by Rhodes and Slachman,[45] and by Keyes and Byman.[21] There is agreement in the conclusions that local Murphree efficiency, overall Murphree efficiency and overall plate efficiency all show a maximum near the center of the liquid concentration range. Furthermore it is pointed out that the viscosity of the liquid also attains a maximum at or near the same point. Results on ethanol-water were essentially duplicated on the same column running isopropyl alcohol-water mixtures by Langdon and Keyes.[26] Rhodes and Slachman[45] also worked with benzene-toluene mixtures, and state that efficiency, as with ethanol-water, is essentially independent of concentration; both of their curves show slight maxima, however. Drickamer and Bradford[11] obtained an essentially linear relation by plotting the logarithm of molal average viscosity versus plate efficiency data for a large number of commercial columns operating on petroleum fractions.

Theoretical considerations indicate that the effect of liquid concentration is twofold. The first factor, brought out in the introductory pages of Drickamer and Bradford's article,[11] is that due to the diffusional film on the liquid side of the gas-liquid interface, plate efficiency should increase as liquid viscosity decreases. The viscosity of a liquid mixture depends upon both the temperature and the composition. Secondly, a noticeable temperature or viscosity effect indicates an appreciable liquid film resistance, since these properties of the liquid are much more greatly affected by temperature and by composition than are the same properties of the vapor. Theo-

retically, the Murphree efficiency is independent of concentration when vapor film controls, but not so when the liquid film resistance is an important factor. This latter point has been developed by Colburn[10] for absorption, and discussed by Gerster, Koffolt and Withrow[15] for distillation.

Column design may affect the relative importance of liquid and vapor films to some extent, which is perhaps a partial explanation of some of the discordant results on different equipment as noted earlier.

Effect of Other Variables

Other operating variables have received scant attention. The conclusion of Kirschbaum and Andrews as to the role played by reflux ratio has already been mentioned. Gay[14] points out that feed plate location is important, stating that the higher the temperature of the feed the lower it should be introduced into the column; this is contrary to theory, and Gay presented no experimental verification of his statement.

Summary

The works of earlier investigators bring out these points: (1) separation efficiency and allowable vapor velocity decrease with decrease in plate spacing, (2) vapor velocity has no marked effect on column efficiency, (3) liquid concentration has been shown to affect column efficiency for the ethanol-water and the isopropanol-water systems, and (4) effects of other important variables have not been determined.

Probably the best summary is given by Keyes and Byman[21] in the sentence:

> Clearcut analysis of the problem of fractional distillation, as derived from well-planned experiments, seems almost impossible because of the complex interaction of the many controlling factors.

STATEMENT OF PROBLEM

This work is divided into several parts, which, in chronological order, are: (1) completion and placing in working order of a 6-inch diameter 12-plate rectifying column and auxiliaries, (2) purification of sufficient benzene and toluene to perform distillation experiments, (3) compilation of thermodynamically consistent data on the enthalpies of liquid, vapor, and of vaporization of benzene and toluene, (4) determination of heat losses from the column, and (5) determination of the effects of several operating variables on the efficiency of rectification.

SUMMARY OF PRESENT EXPERIMENTAL WORK

Equipment

The first part of this work was the completion of the rectifying column and auxiliary equipment. Photographs of various parts are shown in Figure 1, and Figure 2 is a schematic diagram of the unit. A detailed description of the individual parts is given in Part IV of this thesis. Briefly, it consists of:

1. Rectifying column: 6-inch diameter, 12 plates, each plate having two bubble caps arranged so that liquid flows past the caps in series.

2. Reboiler: 40-gallon capacity with internal steam heating coils.

3. Condenser: Double pipe heat exchanger, 15 feet long, made in three sections of concentric 2-inch and 3-inch pipe. Approximately 9 1/2 square feet of heat transfer surface.

4. Bottoms cooler: Identical to condenser.

5. Flow measurement: Rotameters in feed, reflux and products lines, and in main water line to condenser. Orifice meters in by-pass water line to condenser, and in water line to cooler.

6. Temperature measurement: Iron-constantan thermocouples with ice-water cold junction connected to portable potentiometer.

Enthalpy Data for Benzene and Toluene

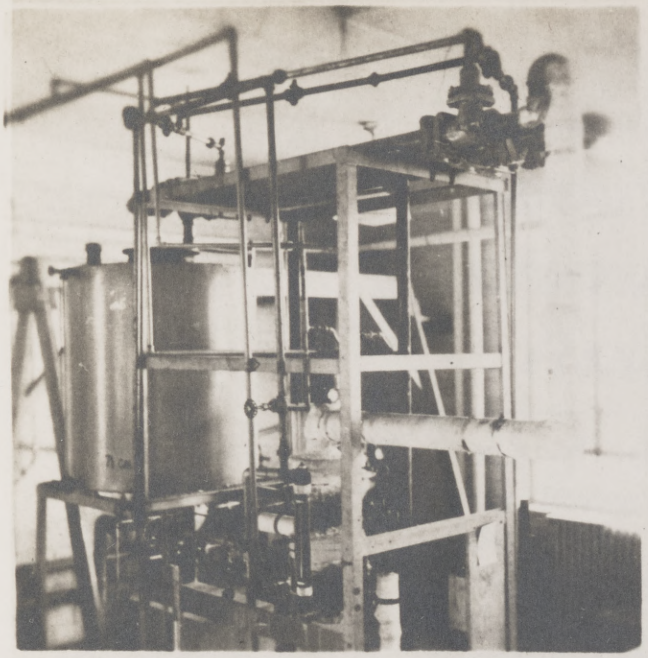
For both benzene and toluene, and for mixtures of the two, thermodynamically consistent enthalpies of the liquid, vapor, and of vaporization were developed from data in the literature.

Fractionation Experiments

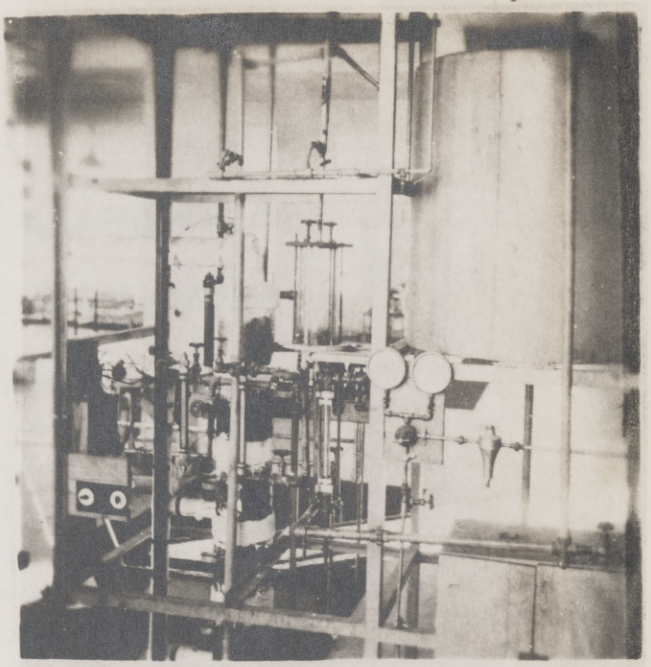
After purification of the benzene and the toluene by reruns and then azeotropic distillations, the following series of runs were made:

1. Fourteen runs at total reflux to determine the heat loss from the column and auxiliaries. The heat loss was calculated as the difference between the sum of the heat given up to the condenser water plus the heat removed in the steam condensate, and the heat input in the steam.

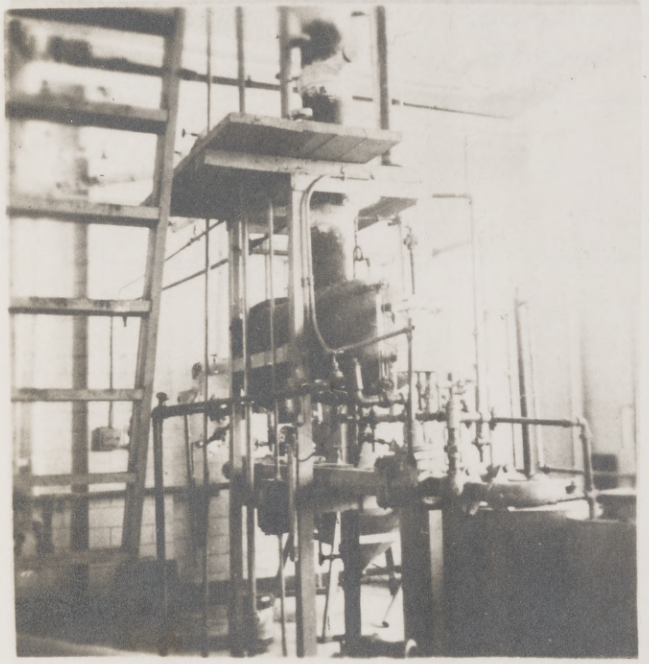
2. Fifteen runs at total reflux to determine the effect of vapor velocity on column performance, and to partially ascertain



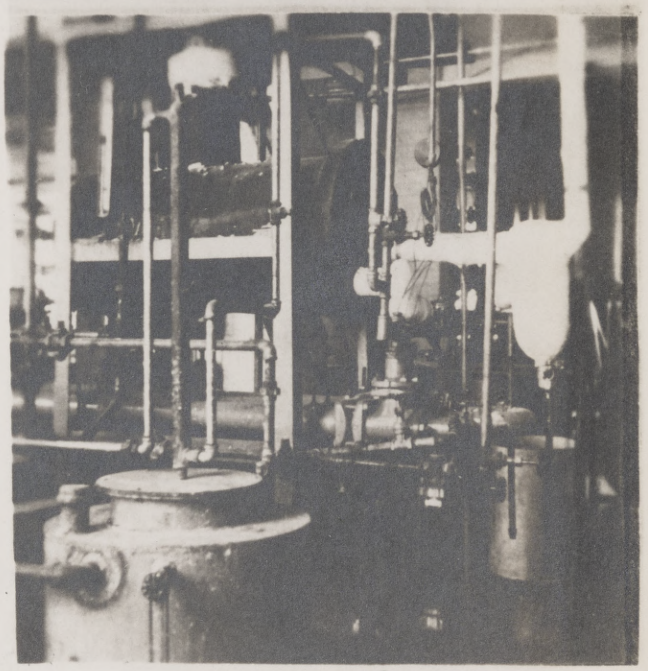
1



2



3



4

FIG. 1

VIEWS OF 12-PLATE COLUMN

1. Condenser, Feed Tank & Column Top
2. Meters, Controls & Reflux Proportioner
3. Reboiler, Tanks & Bottoms Cooler
4. Reboiler, Cooler, Tank & Piping

Fig. 2

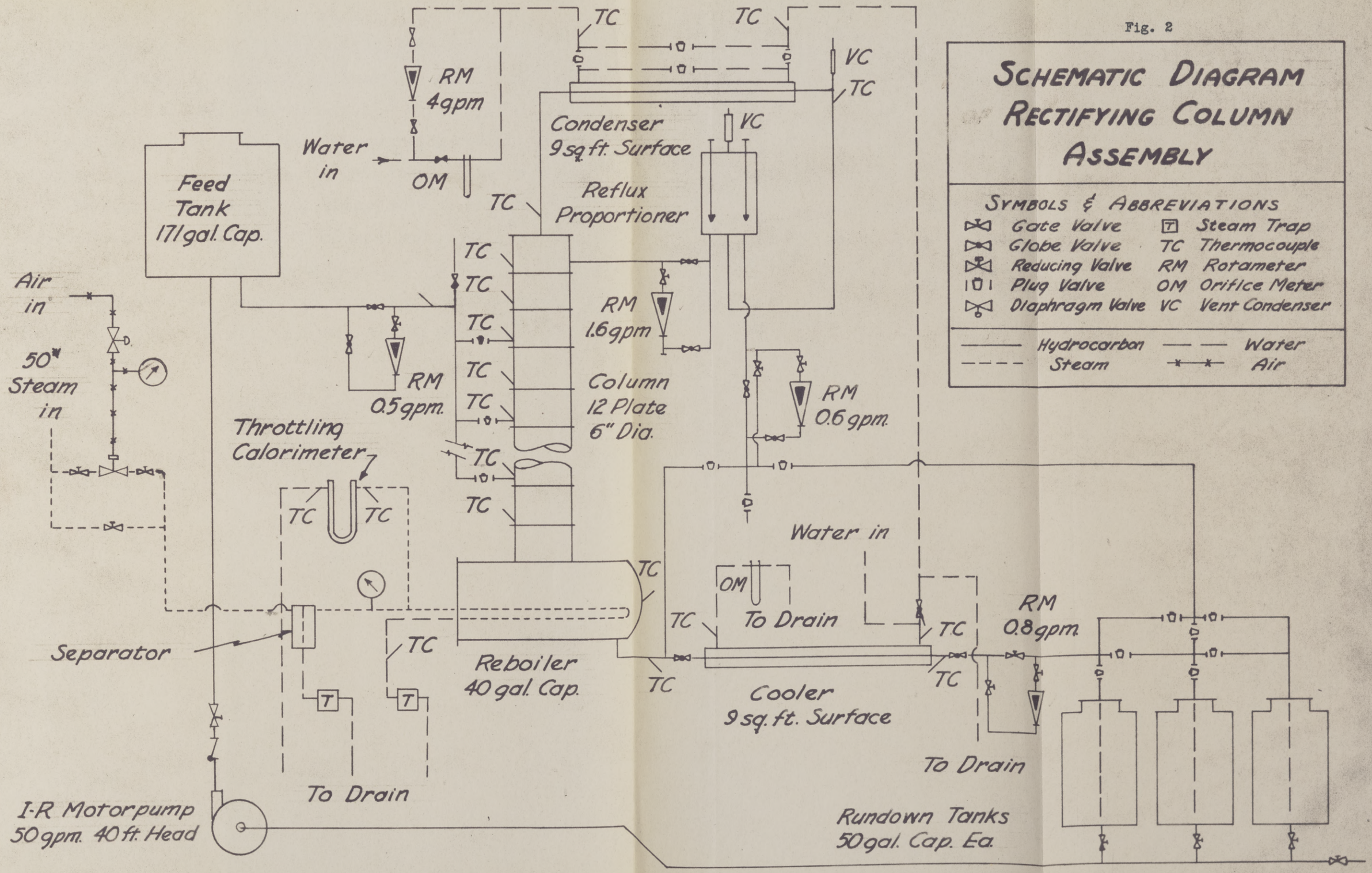
Schematic Diagram Rectifying Column Assembly

Symbols & Abbreviations

Gate Valve		Steam Trap	T
Globe Valve		Thermocouple	TC
Reducing Valve		Rotameter	RM
Plug Valve		Orifice Meter	OM
Diaphragm Valve		Vent Condenser	VC

Hydrocarbon — Water

Steam — Air



the effect of liquid concentration.

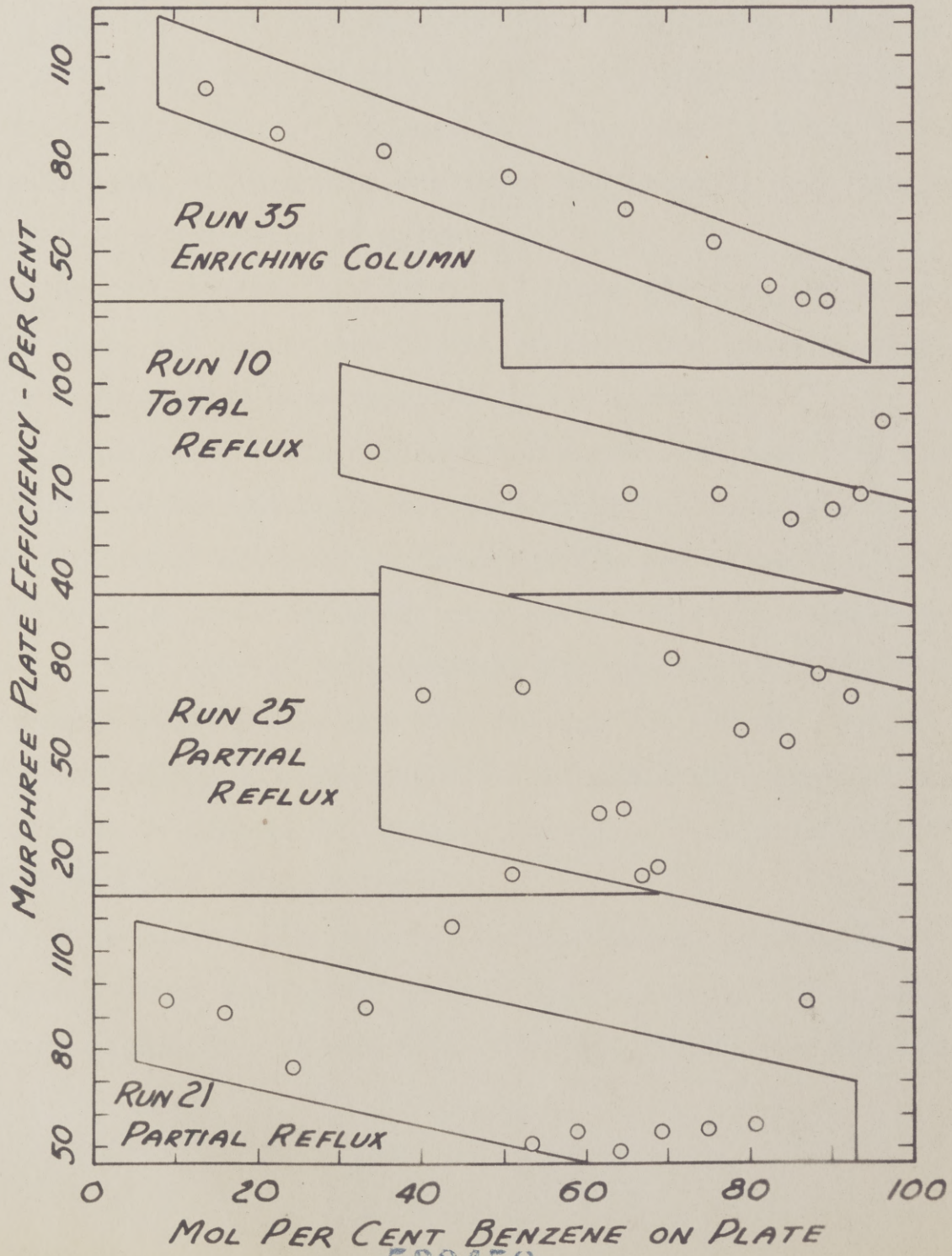
3. Sixteen runs at partial reflux with feed introduced at different plates of the column to study the effects of both concentration and of feed plate location.

4. Two runs as a 12-plate all-enriching column, with the overhead product run continuously into the reboiler as feed.

The equivalent theoretical plates were calculated by the McCabe-Thiele graphical method for each run, and from these the overall column efficiencies were computed. Murphree efficiencies were calculated for several runs, and later converted to the liquid film basis for studying the relative film resistances.

Fig. 3

COLUMN PERFORMANCE
EFFECT OF CONCENTRATION



Notes on Figure 3

Effect of concentration is the obvious starting point for the separation of variables affecting plate efficiency, since it can be shown by data of individual runs (of which operating conditions are always constant).

Runs for each type of operation are included. Run Nos. 10, 21, and 35 are of relatively smooth data, whereas Run No. 25 characterizes many of the partial reflux runs in which the individual plate efficiencies scattered considerably more than occurred in other types of operation.

Reproducibility of individual plate efficiencies is never very high, and smoothness of the present data compare quite favorably with similar data reported in the literature.

Since cold reflux was used throughout and cold feed was introduced to the column in the partial reflux runs, the top plate and feed plate act both as fractionators and as partial condensers. This results in inordinately high efficiencies for these plates.

In the light of the preceding discussion, it is improper to represent plate efficiency data for any one run by a single line since a line is not reproducible. Consequently, straight bands were used to represent the data, excluding consideration of top and feed plate efficiencies.

With due allowance of the factors noted, the slopes of the bands as drawn represent the data best, although for Run 25, scattering of the efficiencies masks the effect of concentration.

II

RESULTS AND CONCLUSIONS

As pointed out in the review of previous work, the operating variables in bubble-tray column rectification are many, and their interrelation is so complex as to make the effect of any one of them extremely difficult to isolate.

The data on all runs were calculated to obtain the overall efficiency (see Part IV). Murphree efficiencies were also computed for several runs of each type: total reflux, partial reflux, and partial reflux with feed introduced into the reboiler.

Effect of Concentration

The effect of concentration was the first variable attempted to isolate. Plots of Murphree efficiency versus concentration for typical runs are given in Figure 3. In general, the plate efficiency-concentration bands are well defined, and all show approximately the same slope, indicating that with decrease in concentration of benzene the efficiency increases. Insofar as can be ascertained, the slopes of the bands for runs both at total reflux and at partial reflux with the feed introduced into the column are the same. Concentration effects in the two total enriching column runs appear to be somewhat greater than in the other types of operation.

It has been noted earlier that efficiency has been tenta-

tively correlated with viscosity for petroleum mixtures in commercial columns. In the case of the benzene-toluene system data on these hydrocarbons,[35,37] indicate that the viscosities are very nearly equal. Furthermore, between 191 and 203 degrees Fahrenheit, which was the usual temperature range in the column, the effect of temperature upon liquid viscosity is small, only about one per cent. Consequently, no viscosity-efficiency effect can be ascertained from these data.

Liquid and Vapor Film Resistances

Griswold[16] developed an efficiency relation based upon liquid film resistance. The Murphree efficiency, which assumes vapor film resistance controlling, is, again:

$$\eta_{M,V} = \frac{y_{n+1} - y_n}{y^*_{n+1} - y_n} = 1 - e^{-f_V A\theta} = 1 - e^{-k} \qquad \ldots 1$$

The corresponding efficiency assuming liquid film resistance controlling is likewise,

$$\eta_{M,L} = \frac{y_{n+1} - y_n}{y^*_{n+1} - y_n} = 1 - e^{-f_L A\theta/m} = 1 - e^{-k/m} \qquad \ldots 2$$

where m is the average slope of the y*-x equilibrium curve between y_n and y_{n+1}, f_V and f_L the vapor and liquid film coefficients respectively, A the effective interfacial area, and θ the

effective time of liquid-vapor contact. From these relations it is obvious that the numerical values of Murphree efficiency must be the same in either case. However, the vapor film efficiency is independent of concentration, while the liquid film efficiency depends upon the slope of the equilibrium curve, m, which decreases as x increases.

The exponential group of equations 1 and 2 is not entirely separable into component variables but is determinable as a whole from experimental plate compositions. However, the slope of the equilibrium curve is known and can be eliminated from the exponential group of equation 2. All the variables of the group should be substantially independent of concentration in any given run.

Figure 4 is a plot of equations 1 and 2 taking constant $(fA\Theta) = 0.92$ for η, and $(0.92/m)$ for η_c; m being the slope of the benzene-toluene equilibrium curve as given in Figure 5. This illustrates the effect of concentration on efficiency where liquid film resistance controls. The exponential function is plotted in Figure 6 for convenience of usage.

A similar procedure was used to develop Figure 7 of experimental efficiencies for typical runs. First, η was found from the experimental data by use of equation 3. Then k was read from Figure 6, and divided by m read from Figure 5 at $y_{avg.}$ for each plate. Next (k/m) was calculated and the efficiency corresponding to it was read from Figure 7 (see Table 13 in Appendix). Thus Figure 4 is of liquid film efficiencies corrected for slope of equilibrium curve according to the relation

Fig. 4

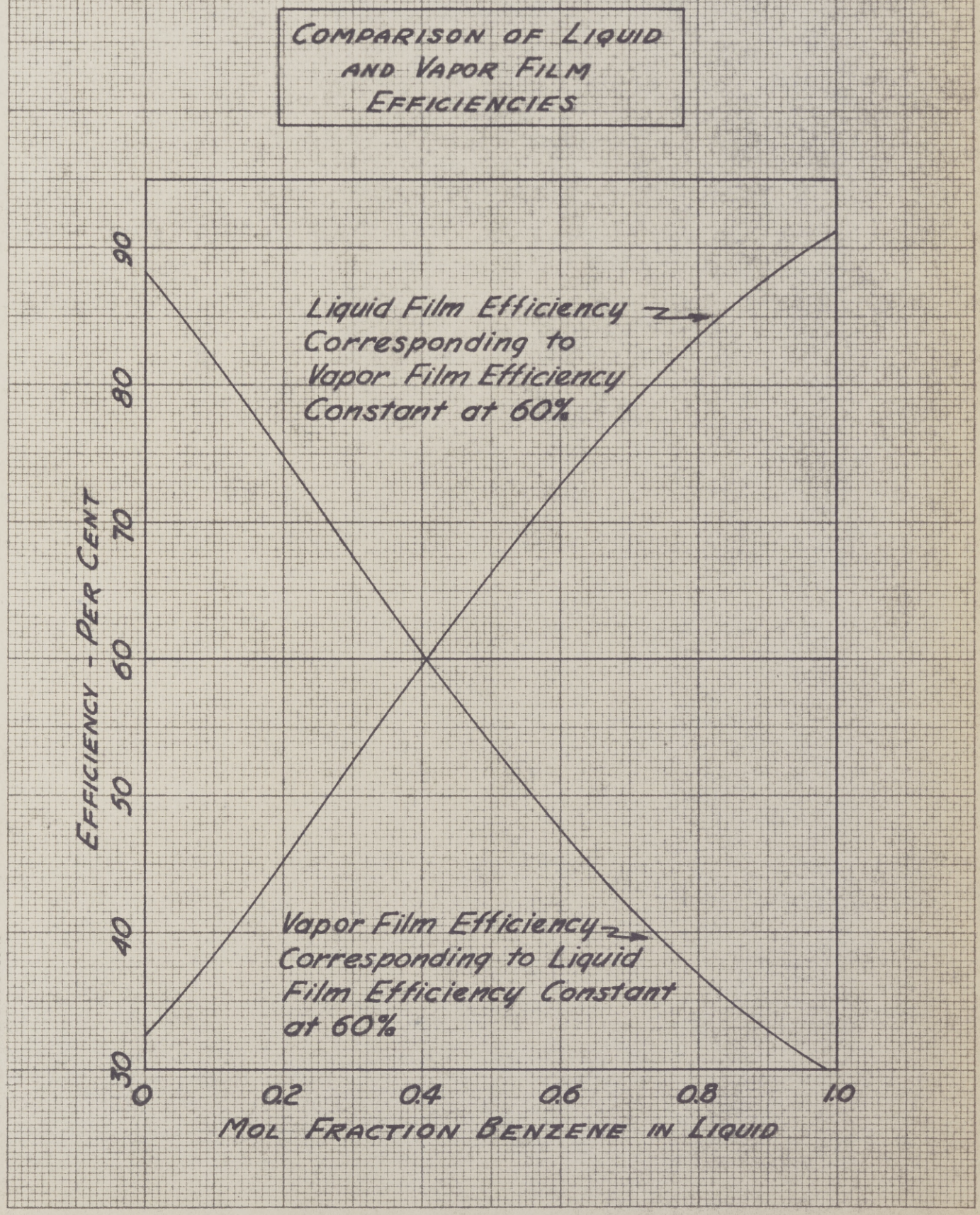
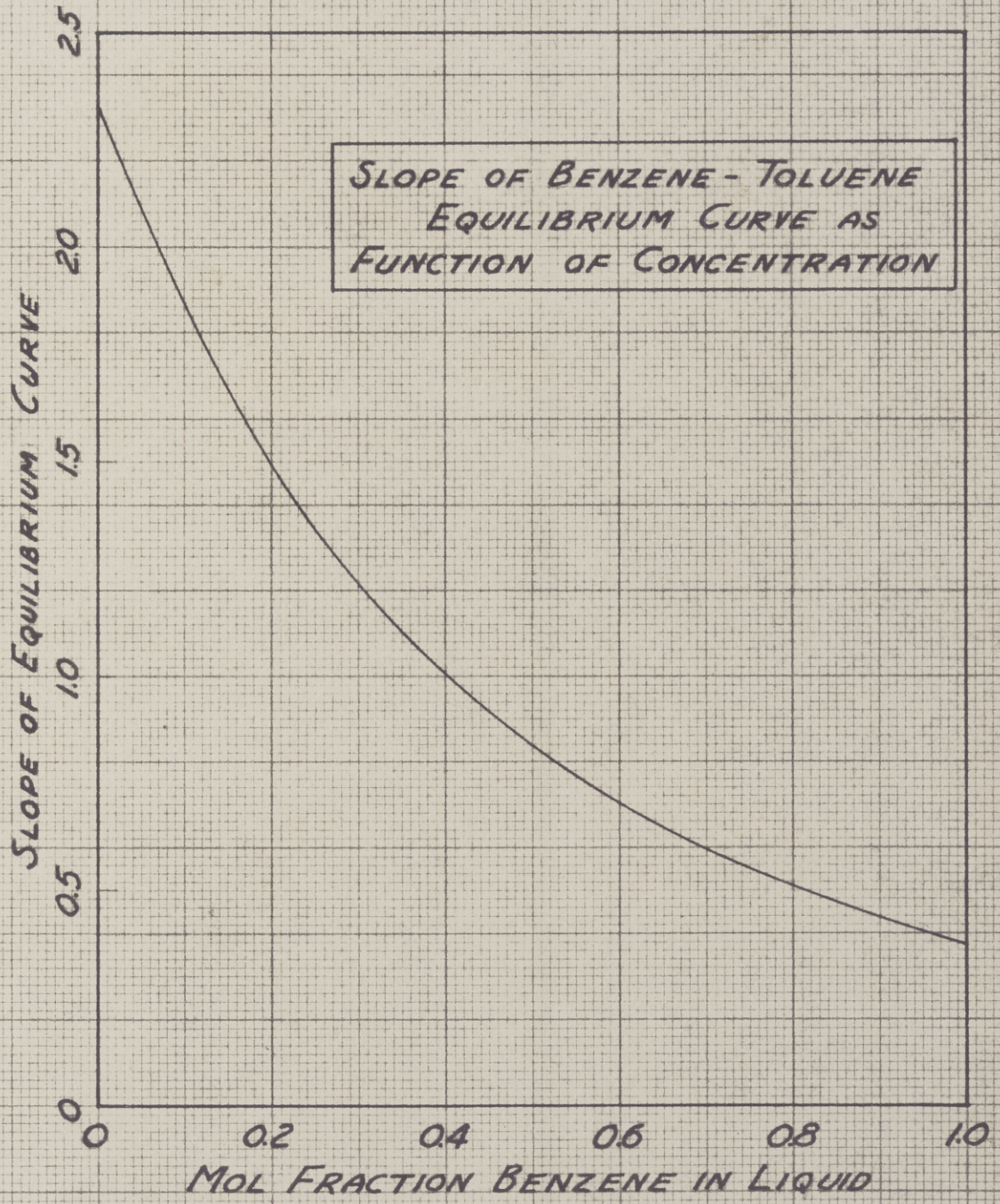


Fig. 5



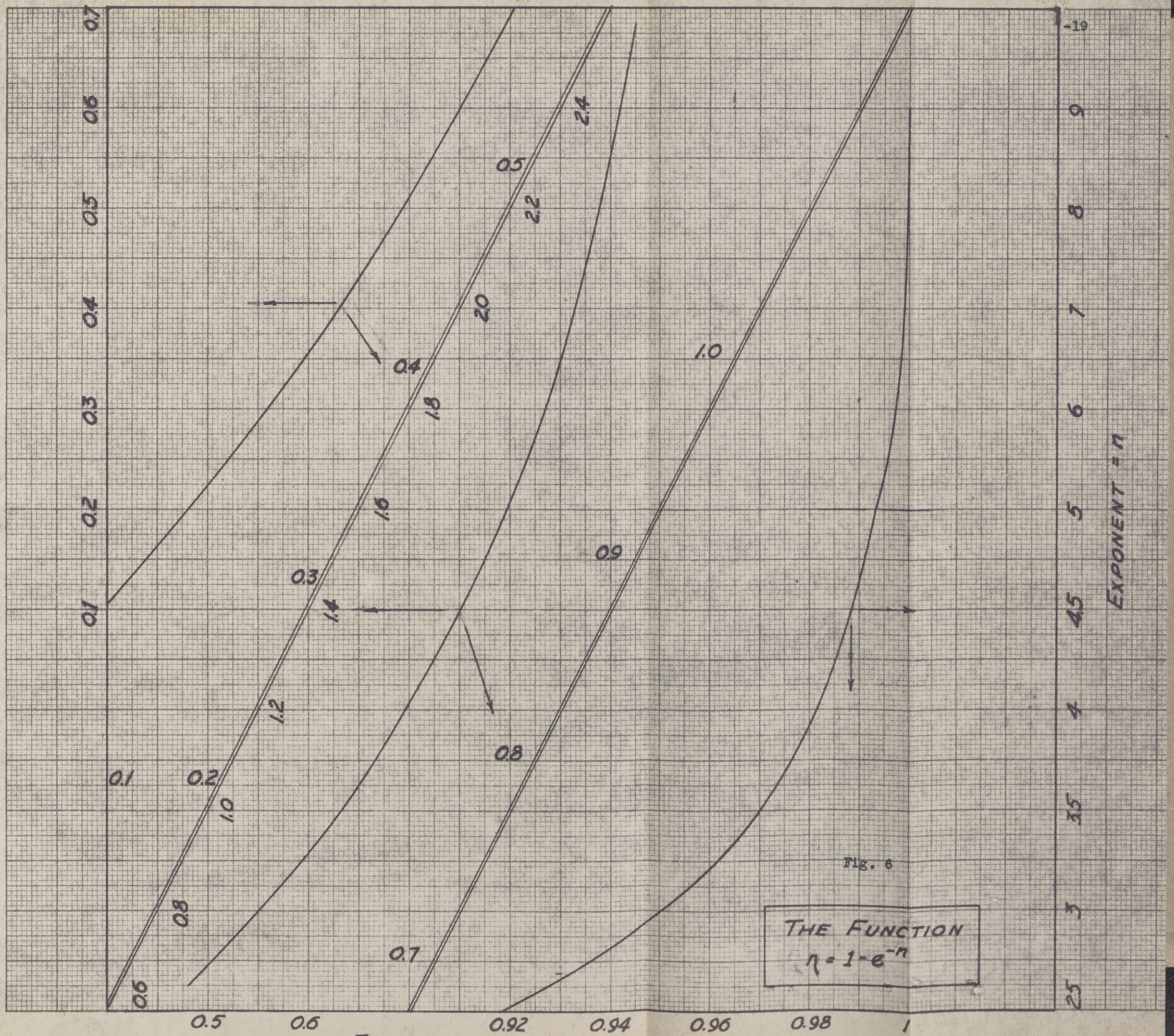
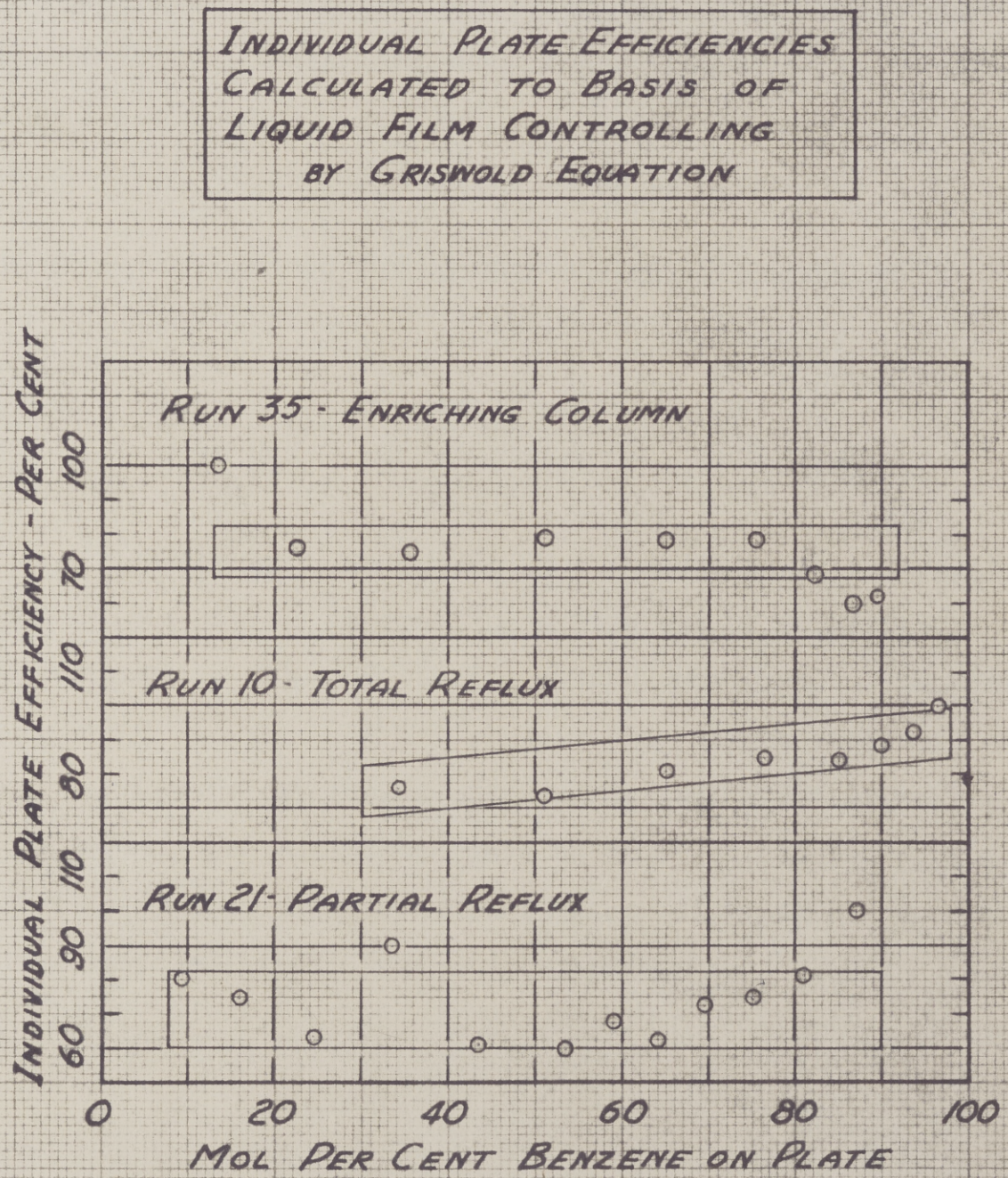


Fig. 6

Fig. 7



$$\eta_c = 1 - e^{-f_L A \Theta} \qquad \ldots 3$$

From the much smaller slopes of the efficiency-concentration bands it is concluded that liquid film controls for the partial reflux runs. Apparently at total reflux both films are important, although why this should be is not known.

Comparison of Overall Column and Murphree Efficiencies

The overall column efficiency,

$$\frac{\text{Equivalent theoretical plates}}{\text{Number of actual plates (12 in this column)}} \qquad \ldots 4$$

is much simpler to compute than is the Murphree efficiency for a multiplate column. Comparison of the two efficiencies on typical runs is given in Table 1 on the next page. From these it is concluded that for the present experiments at least, there is no consistent difference between the two. Further work showed that the Murphree efficiency gave no better consistency in the correlations than did the overall column efficiency; hence the latter was used in all remaining correlations.

TABLE 1

COMPARISON OF OVERALL AND MURPHREE EFFICIENCIES

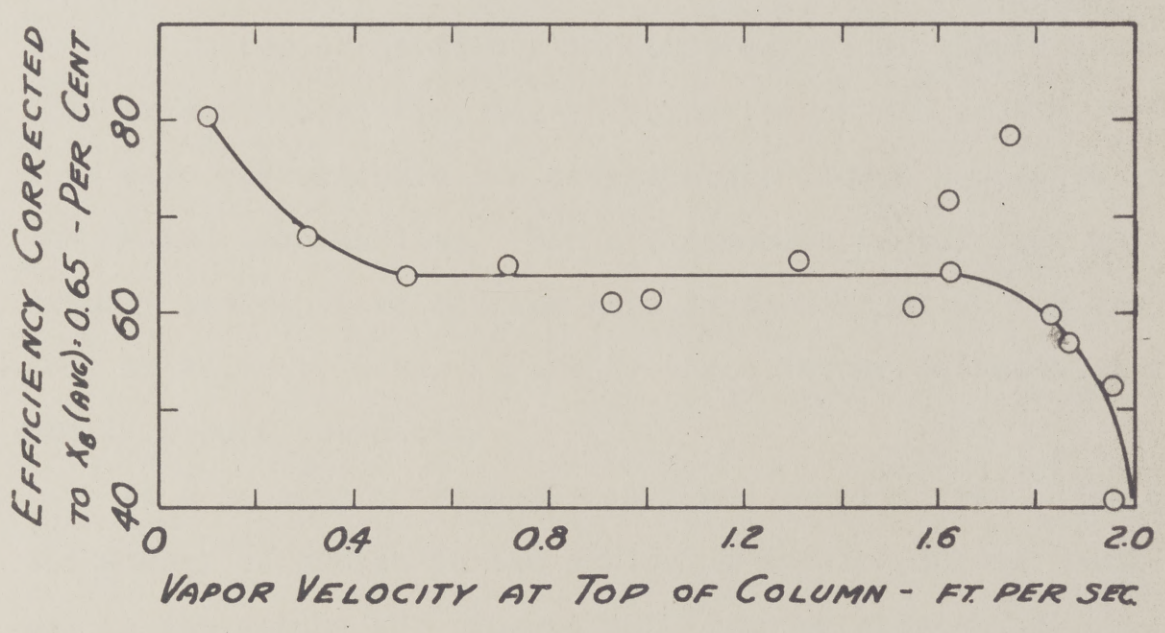
Run Number	5	6	7	10	21	24	25	35
Murphree Eff.								
Plate 12					94.3		68.6	34.9
11		96.0			56.6		75.7	35.1
10		69.8			55.2		54.6	40.8
9		62.5			53.7		57.6	53.3
8	95.3	50.6	90.5	87.5	48.5		80.0	63.2
7	65.0	44.8	68.2	65.8	55.5	65.2	15.6	72.9
6	65.2	38.1	58.3	60.4	51.5	56.7	13.3	78.2
5	66.7	39.4	57.3	57.8	118.0	61.5	33.3	86.7
4	63.1	38.2	43.7	65.9	92.7	59.0	32.7	100.0
3	59.9	41.3	40.6	65.3	74.2	57.4	71.8	
2	62.2	43.6	39.5	66.7	90.2	62.5	13.0	
1	70.6	57.2	55.3	79.8	95.5	75.9	69.4	
Avg. Murphree Efficiency	68.7	52.9	56.7	68.6	73.8	62.6	48.8	62.8
Overall Col. Efficiency	58.6	48.9	54.3	62.9	80.0	51.0	44.3	Indet.

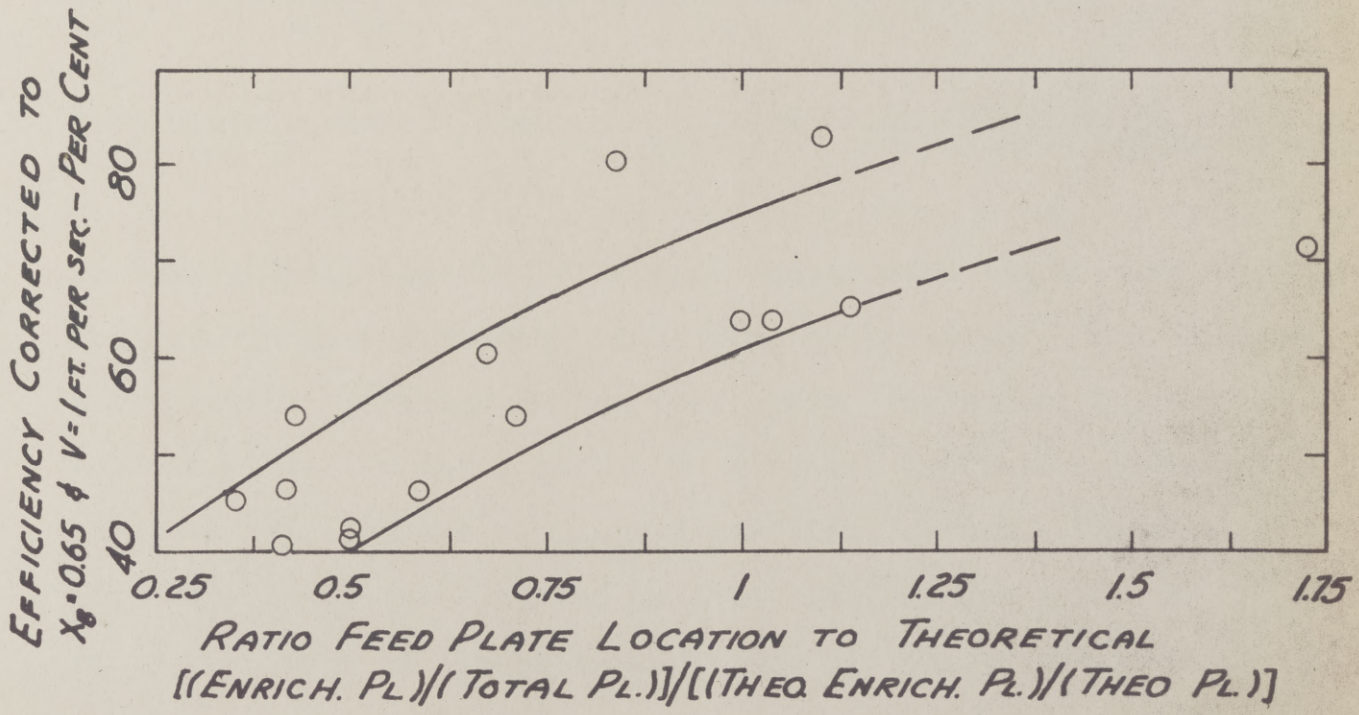
Effect of Vapor Velocity

In order to establish the role of vapor velocity it was necessary to assume that the effects of this variable and of concentration are algebraically additive. Therefore, the efficiencies calculated for the total reflux runs were corrected to a common concentration ($x_{B,W} = 0.65$), which was selected because it was the average value for many of the runs, as shown in the summary of calculations. The efficiencies corrected for concentration were then plotted as in Figure 8, which indicates that over the middle half of the vapor velocity range from lowest practicable operating conditions to flooding,

Fig. 8

COLUMN PERFORMANCE
EFFECTS OF VAPOR VELOCITY
AND FEED PLATE LOCATION





vapor velocity has no detectable effect on efficiency. At very low velocities the efficiency was found to increase somewhat, while it markedly decreased at velocities approaching the flood point.

Effect of Feed Plate Location

The point of introduction of the feed was varied over quite a number of positions in different runs. Again assuming all variables additive, the efficiencies of all partial reflux runs were corrected to the same average liquid concentration by the method used earlier. The efficiencies of two runs at extremely low vapor velocities were further adjusted for the effect of velocity using Figure 8; these latter corrections were small in both instances.

As a measure of actual feed plate location the ratio of the number of plates in the enriching section to the total number in the column was chosen, which can be written

$$\text{Feed plate location index} = \frac{\text{Plates in enriching section}}{\text{Plates in enriching + stripping sections}}$$

In this calculation the feed plate was counted twice, that is, included in both enriching and stripping sections. To determine a theoretical location of the feed plate, the number of theoretical plates at total reflux to give the overhead and bottoms products was computed by the McCabe-Thiele method. The feed composition was then located on this diagram, and again the ratio of number of plates in enriching section to total plates

calculated. These two ratios, actual and "theoretical" location of feed plate, were then divided, one by the other, and the resulting ratio plotted against corrected efficiency as shown in Figure 8. The points on this plot scatter quite markedly, so here, too, a band correlation was made. This graph indicates that the nearer the actual is to the "theoretical" location of the feed plate when the feed is introduced above the "theoretical" location, the higher the efficiency. Cold liquid feed was used in all runs.

However, when the feed was introduced below the "theoretical" location, efficiency of operation as measured by plate efficiency actually gave improved column performance. This is further substantiated by the two runs in which the apparatus was used as an all-enriching column with the overhead product recycled to the reboiler as feed. In these runs the bottoms product was purer toluene than the McCabe-Thiele method of calculation indicates that it is possible to obtain, which phenomenon can be designated as very high efficiency. No explanation is offered for this apparent anomaly, but the trend is evident in a sufficient number of runs and the reproducibility was sufficiently good so that it may not be ascribed to experimental error.

Effect of Heat Loss

From a study of the calculations on the partial reflux runs it is apparent that the internal reflux produced by heat loss becomes an important factor only when the vapor velocity is low. The quantity of vapor condensed by heat loss per unit time is

essentially constant in any one section of the column. At low vapor rates this quantity appreciably changes the magnitude of the rate of flow; at higher rates heat-loss condensate is only a small fraction of the reflux from the condenser. Below the feed plate in all cases, the effect of heat-loss condensate on (L/V) is negligible, but at low rates it becomes significant above the feed plate. As shown in the sample calculation for Run 29, given in the Appendix, the slope of the enriching line was corrected for heat loss at intervals of four plates by an enthalpy balance.

At medium to high vapor velocities, therefore, the column may be considered to be substantially adiabatic.

Miscellaneous

No correlation was attempted between reflux ratio and efficiency. This much might be said: with decreasing values of (L/V) the efficiency seems to increase somewhat, although the points scatter too greatly to show conclusively any effect.

As shown in Table 10, in all but two runs the feed plate liquor was richer in benzene than was the feed stock. Also, the "q line"[48] method of locating the intersection of the operating lines gave results that were consistently low; i.e., the intersection given by the "q line" and the enriching line was at a lower value of x, from 10 to 15 per cent units, than was the intersection determined by the feed plate composition and the enriching line. The "q-line" calculation of feed plate composition is valid only at theoretical minimum reflux ratio,

as pointed out by Underwood.[55]

Another general conclusion that can be drawn from this work is that the column tends to send overhead a product that is very nearly pure benzene, no matter how the operating conditions are varied.

Summary

Briefly, the results of this work show:

1. The effect of vapor velocity upon efficiency for this column when operating on benzene and toluene at least, is negligible except at the extreme ends of the vapor velocity range.

2. The separation efficiency increases approximately with the concentration of toluene in the plate liquid and independently of other variables, as required by a controlling liquid film resistance.

3. The feed should be introduced at or below the theoretical location to obtain maximum efficiency. When the feed is introduced too high in the column the efficiency decreases; when too low, it apparently increases.

4. Variation in reflux ratio shows no marked effect on efficiency.

5. For partial reflux runs the liquid film controls; for total reflux both films are important.

These conclusions are of significance in operations where a fractionating column has an inadequate number of plates to produce high purity or ultimate separation in both overhead and bottoms products, and where cold feed is used. This is a case of common industrial practice.

III

THERMAL PROPERTIES OF BENZENE AND TOLUENE

Comprehensive fractional distillation calculations require accurate thermal properties of the components and of their mixtures. A literature survey was made to determine the best thermodynamically consistent values of the properties for benzene and toluene: enthalpy of liquid, enthalpy of vapor, and enthalpy of vaporization, at various temperatures.

BENZENE

A complete compilation for benzene has been made by the National Bureau of Standards[13] over the temperature range 0° to 130° C. Consequently all that needed to be done with these data was to convert the metric units (in which they were reported) to English engineering units. These data are presented in Table 2, and graphically in Figure 9.

TOLUENE

Only random, and for the most part incomplete, data are available for toluene. Vapor pressure values are reported by Krase and Goodman[25] and by Barker,[2] as well as in the International Critical Tables.[34] Data from these sources were smoothed by a plot of reciprocal of absolute temperature versus log of vapor pressure, and the vapor pressure values used were those read from this graph.

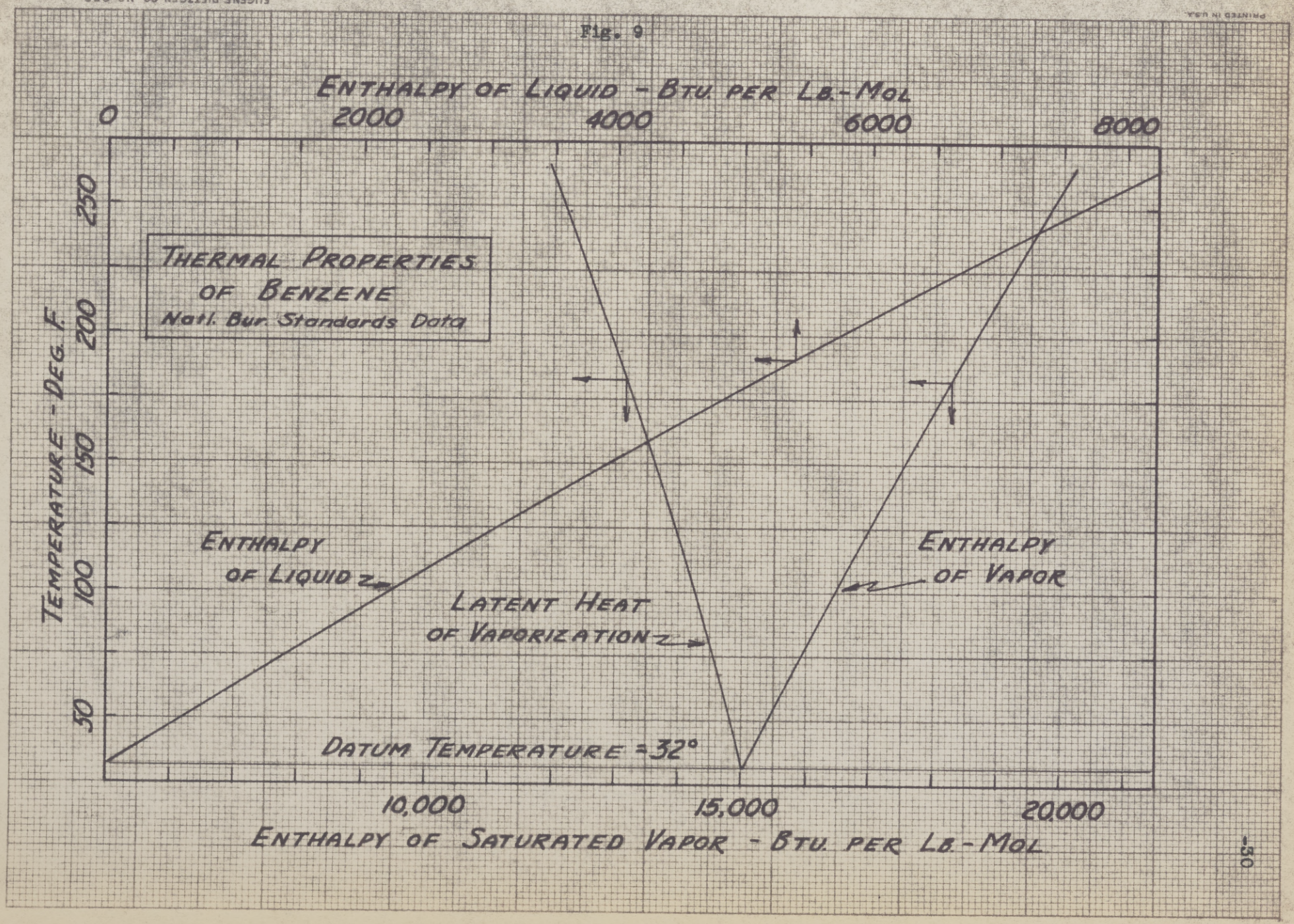
Apparently there is only one reported determination of the latent heat of vaporization of toluene; that by Mathews[29]

TABLE 2

PROPERTIES OF BENZENE

Temp °F.	Vapor Press. PSIA	Specific Volume Cu.ft.per lb. Sat. Liq.	Specific Volume Cu.ft.per lb. Sat. Vapor	Heat Btu per Lb. of Sat. Liq.	Heat Btu per Lb. of Vaporization	Heat Btu per Lb. of Sat. Vap.	Specific Volume Cu.ft. per mol Sat. Liq.	Specific Volume Cu.ft. per mol Sat. Vapor	Heat Btu per Mol of Sat. Liq.	Heat Btu per Mol of Vaporization	Heat Btu per Mol of Sat. Vapor
32	0.513	1.779	141.02	0	192.3	192.3	1.390	11,015	0	15,021	15,021
50	0.880	1.800	79.76	7.436	189.7	197.2	1.406	6,230	580.8	14,817	15,403
68	1.444	1.823	50.08	14.96	187.1	202.0	1.424	3,912	1,169	14,614	15,778
77	1.815	1.834	39,11	18.75	185.7	204.5	1.433	3,055	1,465	14,505	15,973
86	2.287	1.845	32.53	22.59	184.4	206.9	1.441	2,541	1,764	14,403	16,160
104	3.356	1.867	21.81	30.32	181.5	211.8	1.458	1,704	2,368	14,177	16,544
122	5.202	1.891	15.06	38.17	178.7	216.8	1.477	1,176	2,981	13,958	16,934
140	7.516	1.917	10.71	46.15	175.6	221.7	1.497	836.6	3,605	13,716	17,321
158	10.588	1.943	7.778	54.23	172.6	226.8	1.518	607.6	4,236	13,482	17,715
176	14.576	1.967	5.777	62.45	169.4	231.9	1.536	451.2	4,880	13,232	18,114
194	19.654	1.992	4.372	70.79	166.1	236.9	1.556	341.5	5,529	12,974	18,504
212	26.004	2.021	3.367	79.30	162.7	242.0	1.579	263.0	6,194	12,708	18,903
230	33.810	2.052	2.630	87.95	159.3	247.2	1.603	205.4	6,870	12,443	19,309
248	43.290	2.082	2.082	96.77	155.6	252.4	1.626	162.6	7,559	12,154	19,715
266	54.640	2.116	1.674	105.71	151.9	257.6	1.653	130.7	8,257	11,865	20,121

Fig. 9



at its normal boiling point. Of necessity, recourse was made to several methods for estimating such properties. Othmer's "reference substance" correlation of latent heat with vapor pressure[40] (which may be derived from the Clausius-Clapeyron equation) is:

$$\log P = (M\lambda / M'\lambda')\log P' + C \quad \ldots 5$$

where the P terms refer to vapor pressures, and the ($M\lambda$) terms to molar latent heats of vaporization. Choosing benzene as the reference substance, a plot of log P versus log P' was made. The slope of the straight line obtained on this graph gave the ratio of the latent heats: 1:1.105. This enabled a table of values for toluene to be calculated from the known latent heats of benzene.

A second method for estimating latent heats of vaporization from vapor pressure data, also based on the Clapeyron equation, has been proposed by Fallon and Watson.[56] Their relation, which corrects for the non-ideality of the vapor phase, is

$$M\lambda = \frac{\mu RT^2}{P}(dP/dt) \quad \ldots 6$$

where standard symbols are used. A series of values of the molar latent heats of vaporization for toluene was calculated from this equation.

The values from Equation 5 were found to be in much better

agreement with the single reported experimental value than those calculated by Equation 6. The value given by Equation 5 for 110°C. is less than 1/2 of 1 per cent low. These calculated values were assumed to be correct.

Data on the heat capacity of liquid toluene are given by Kelley,[20] Smith and Andrews,[52] International Critical Tables,[36] and Williams and Daniels.[57] The agreement among the first three sets of data is quite good, but those of Williams and Daniels are not at all concordant with the others; this is perhaps due to the fact that the last-named measurements were made on a "super-dry" material. As a further check, several values were calculated from Watson's empirical equation

$$C_p = [(0.355 + 0.128 \times 10^{-2}(^\circ API) + (0.503 + 0.117 \times 10^{-2}(^\circ API)) 10^{-3} t] \times [0.05K - 0.41] \quad \ldots 7$$

where

$$K = \text{UOP characterization factor} \left[\frac{(\text{b.p. in } ^\circ R)}{\text{sp. g. at } 60^\circ F.}\right]^{1/3}$$

$^\circ API$ = density on API scale = 30.8 for toluene.

The calculations gave values that were consistently lower than those determined experimentally, although the slope of the straight line on the plot of molar heat capacity versus temperature was in good agreement with the data.

Specific heat data for toluene vapor are reported by Stull,[54] (from calculations based on spectroscopic data), and by Montgomery and DeVries,[31] from experimental work. The agreement is

quite good between these two sets of values.

In order to obtain thermodynamic consistency among latent heat of vaporization, heat capacity of liquid, and heat capacity of vapor, recourse was had to the basic equation

$$(MC_p)_{liq}(t_2 - t_1) + (M\lambda)_{t_2} = (MC_p)_{vap}(t_2 - t_1) + (M\lambda)_{t_1} \quad \ldots 8$$

where standard symbols are used. On the assumption that the latent heats of vaporization were probably the most accurate of all values and that experimental heat capacities of the liquid were essentially correct, the specific heats of the vapor reported by Stull was reduced slightly (see Figures 11 and 12) so as to give consistent results when substituted in Equation 8. The results were used to calculate the molal enthalpies of toluene, and these are given in Table 3 and Figure 10.

BENZENE-TOLUENE MIXTURES

The next step was to prepare an enthalpy concentration chart for benzene-toluene mixtures. The general equation used in calculating the enthalpy of a liquid mixture is

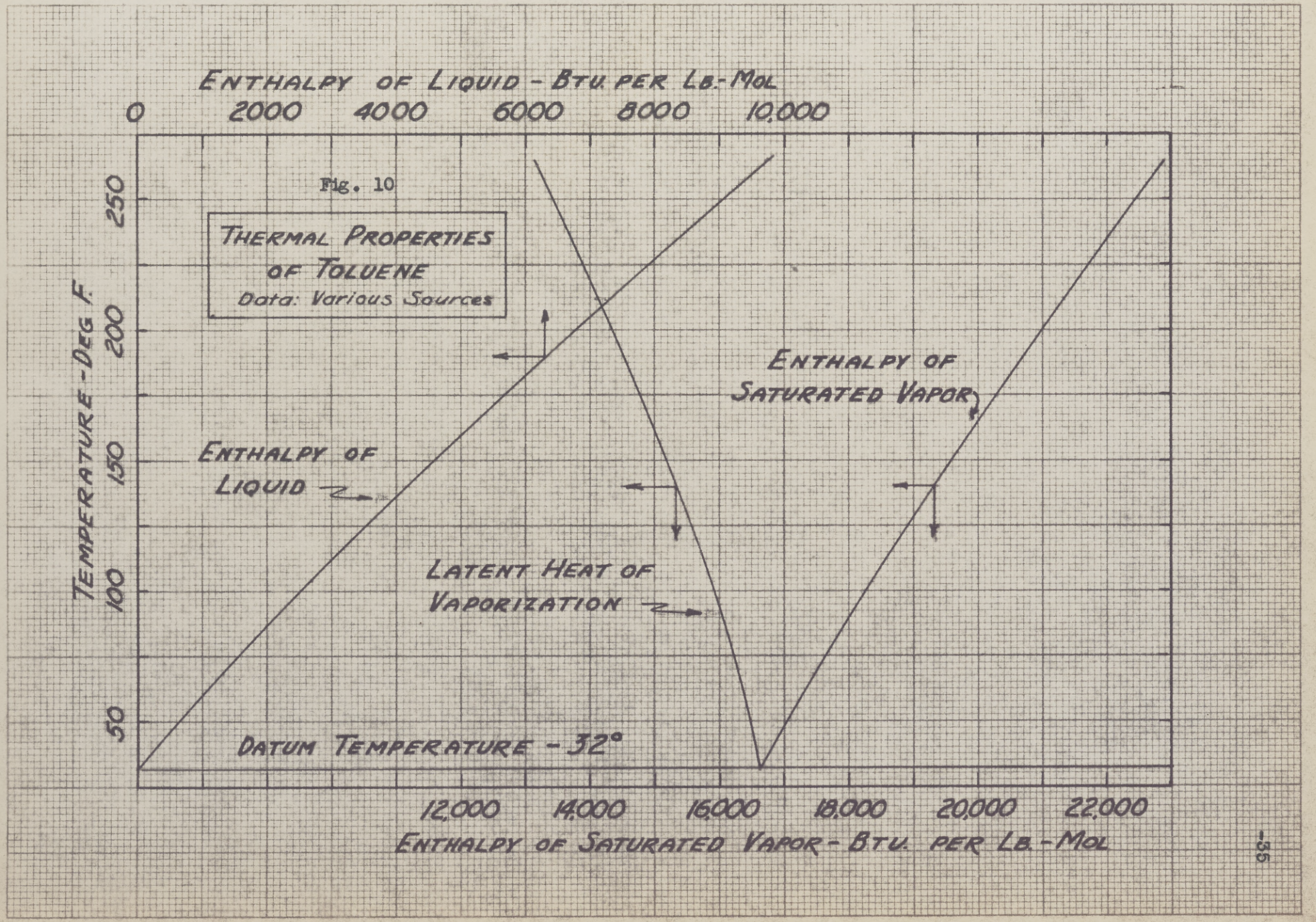
$$H_{mix} = n_A H_A + n_B H_B \quad \ldots 9$$

where H_A and H_B are taken at the temperature of the mixture. This neglects the heat of mixing, but this quantity is small, as shown by the data of Baud[3] reported in I.C.T.[37] Enthalpies of

TABLE 3

PROPERTIES OF TOLUENE

Temp. °F.	Vapor Press. PSIA	Vapor Press. Atm.	Molar Latent Heat of Vap. Btu/ Mol.	Molar Heat Capacity Btu/Mol.(°F) of Liquid	Molar Heat Capacity Btu/Mol.(°F) of Vapor	Heat Btu per Mol. of Sat. Liq.	Heat Btu per Mol. of Vapor-ization	Heat Btu per Mol. of Sat. Vap.
32	0.137	0.0093	16,620	35.70	23.16	0	16,620	16,620
50	0.257	0.0175	16,400	36.60	23.72	650	16,400	17,050
68	0.441	0.030	16,130	37.50	24.32	1,320	16,130	17,450
86	0.71	0.0483	15,920	38.42	24.88	2,000	15,920	17,920
104	1.07	0.0727	15,670	39.33	25.47	2,700	15,670	18,370
122	1.79	0.122	15,420	40.26	26.02	3,420	15,420	18,840
140	2.69	0.183	15,200	41.18	26.58	4,150	15,200	19,350
158	3.91	0.266	14,900	42.08	27.16	4,900	14,900	19,800
176	5.60	0.381	14,630	43.00	27.72	5,670	14,630	20,300
194	7.80	0.531	14,330	43.93	28.28	6,440	14,330	20,770
212	10.68	0.733	14,080	44.86	28.83	7,250	14,080	21,330
230	14.68	0.998	13,780	45.80	29.38	8,070	13,780	21,850
248	19.1	1.30	13,430	46.72	29.92	8,890	13,430	22,320
266	26.0	1.77	13,120	47.62	30.46	9,750	13,120	22,870



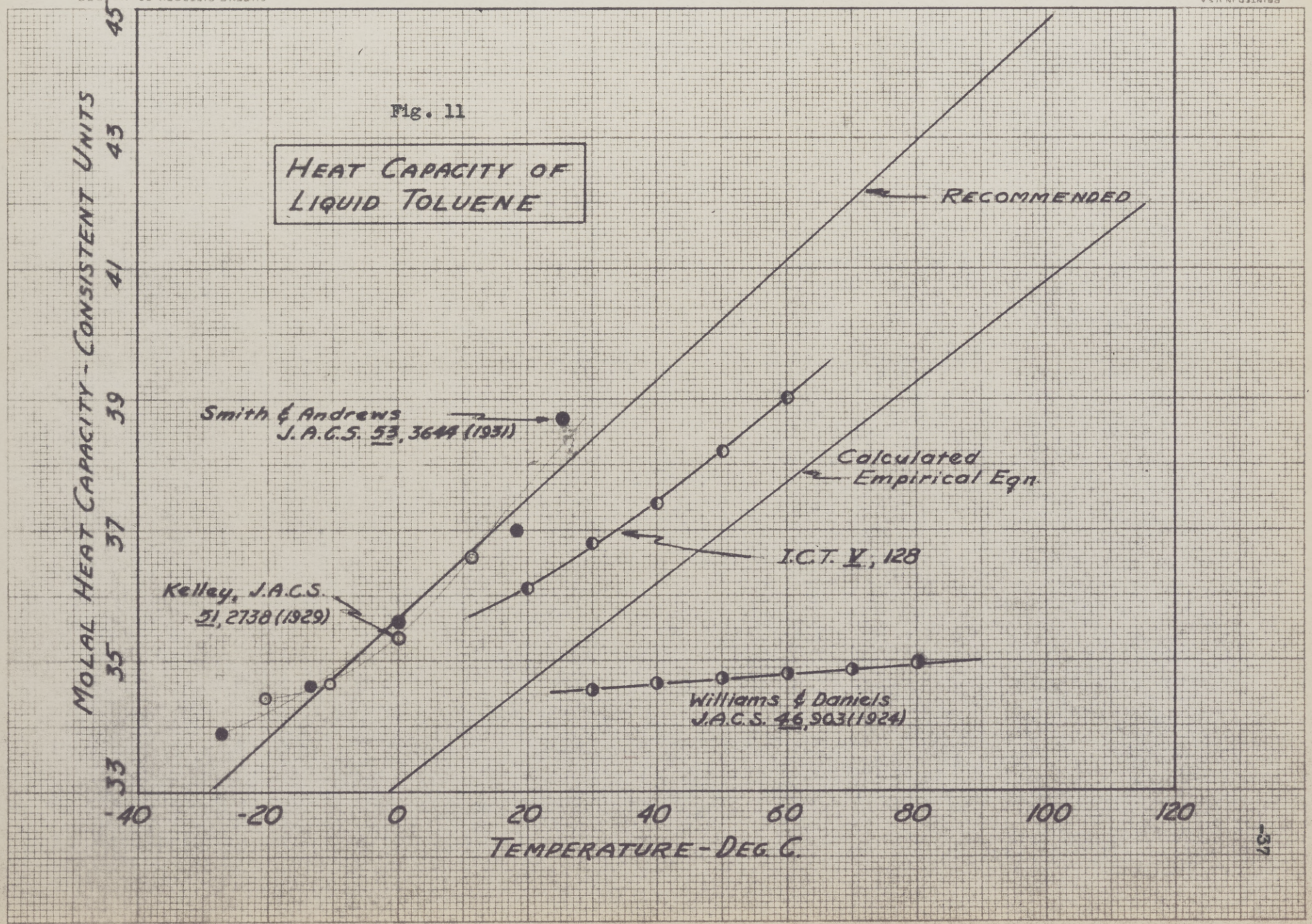
vapor mixtures were obtained by drawing a straight line between the enthalpies of the two pure vapors. Tie lines can be drawn in by using the vapor-liquid equilibrium data of Rosanoff, Bacon and Schulze[49] which are reproduced in convenient nomographic form by Griswold, Andres and Klein.[17] The results of these calculations as 740, 760 and 780 mm. isobars are shown in Table 4 and Figure 13.

TABLE 4

ENTHALPY CONCENTRATION DATA FOR LIQUID BENZENE-TOLUENE MIXTURES

Mol % Benzene	0	20	40	60	80	100
Pressure	Enthalpies, as Btu/lb. mol. of saturated vapor above 32°F.					
740 mm.	8005	7115	6370	5740	5270	4860
760 mm.	8090	7198	6457	5830	5345	4900
780 mm.	8165	7270	6535	5920	5400	4960

Fig. 11



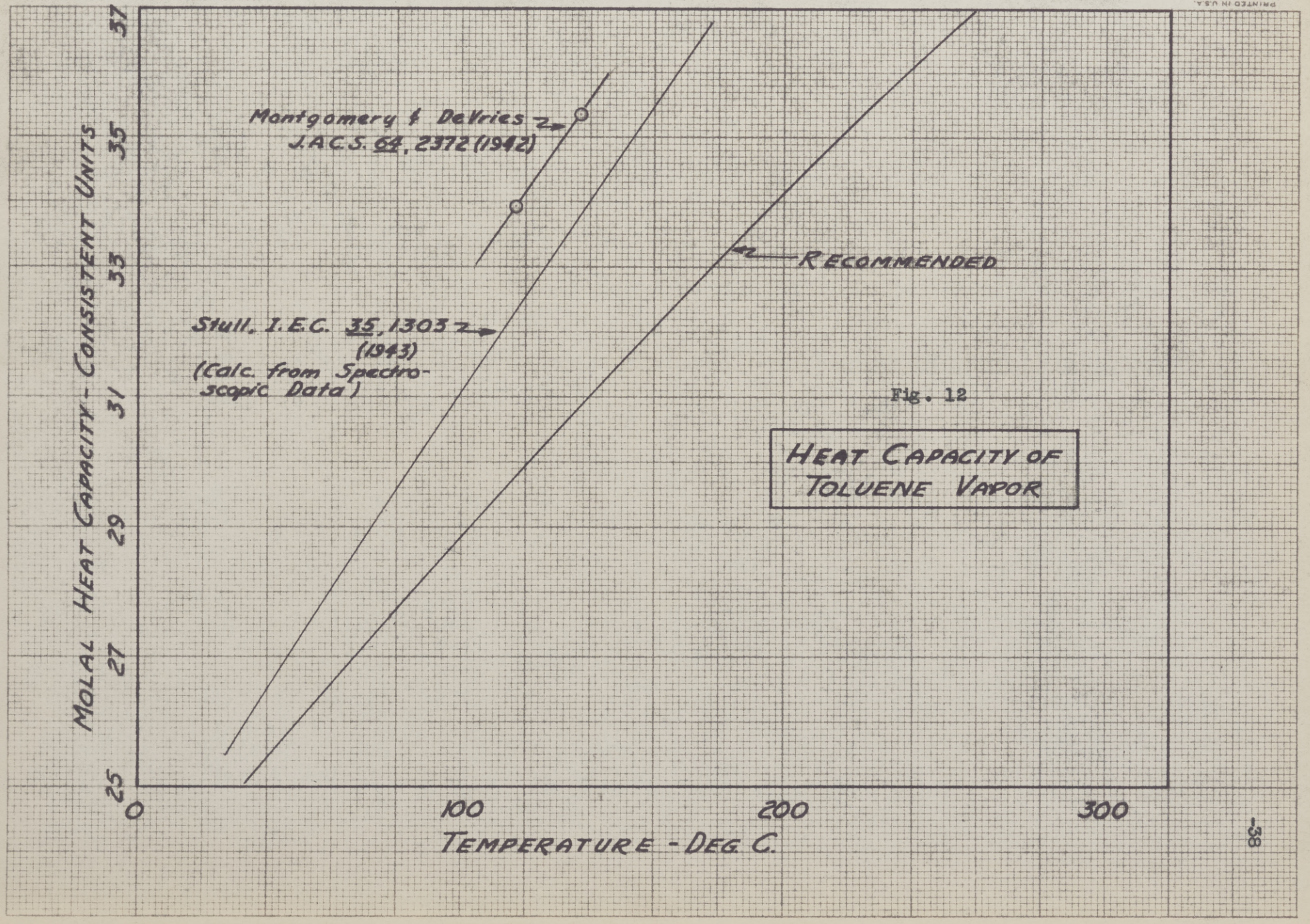
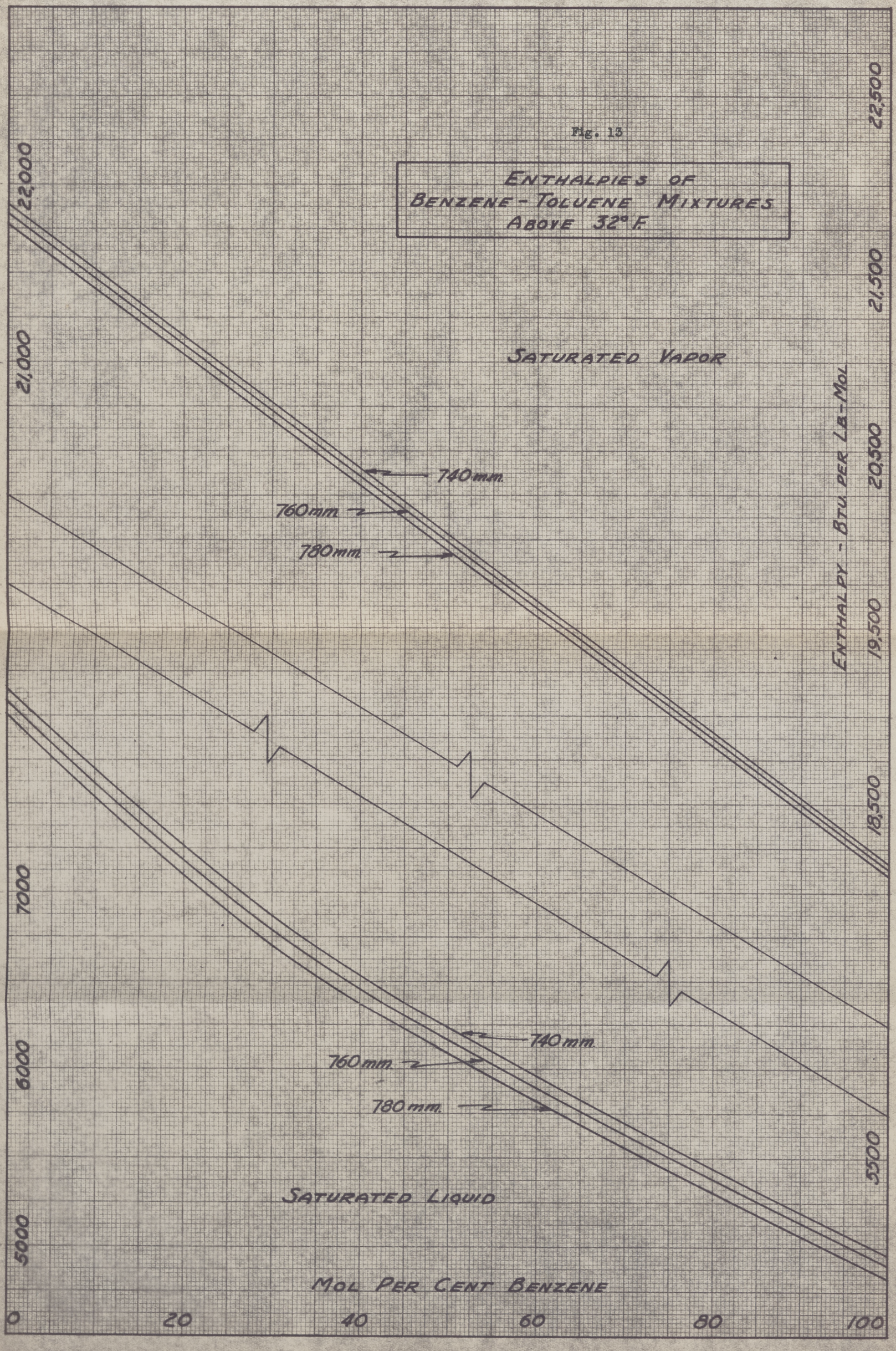


Fig. 12

Fig. 13

Enthalpies of Benzene - Toluene Mixtures Above 32° F.



IV

EXPERIMENTAL WORK

EQUIPMENT

The fractionating column was constructed in the departmental shops. All parts were made of steel unless otherwise noted below. The column and auxiliary equipment (Figures 1 and 2) consist of:

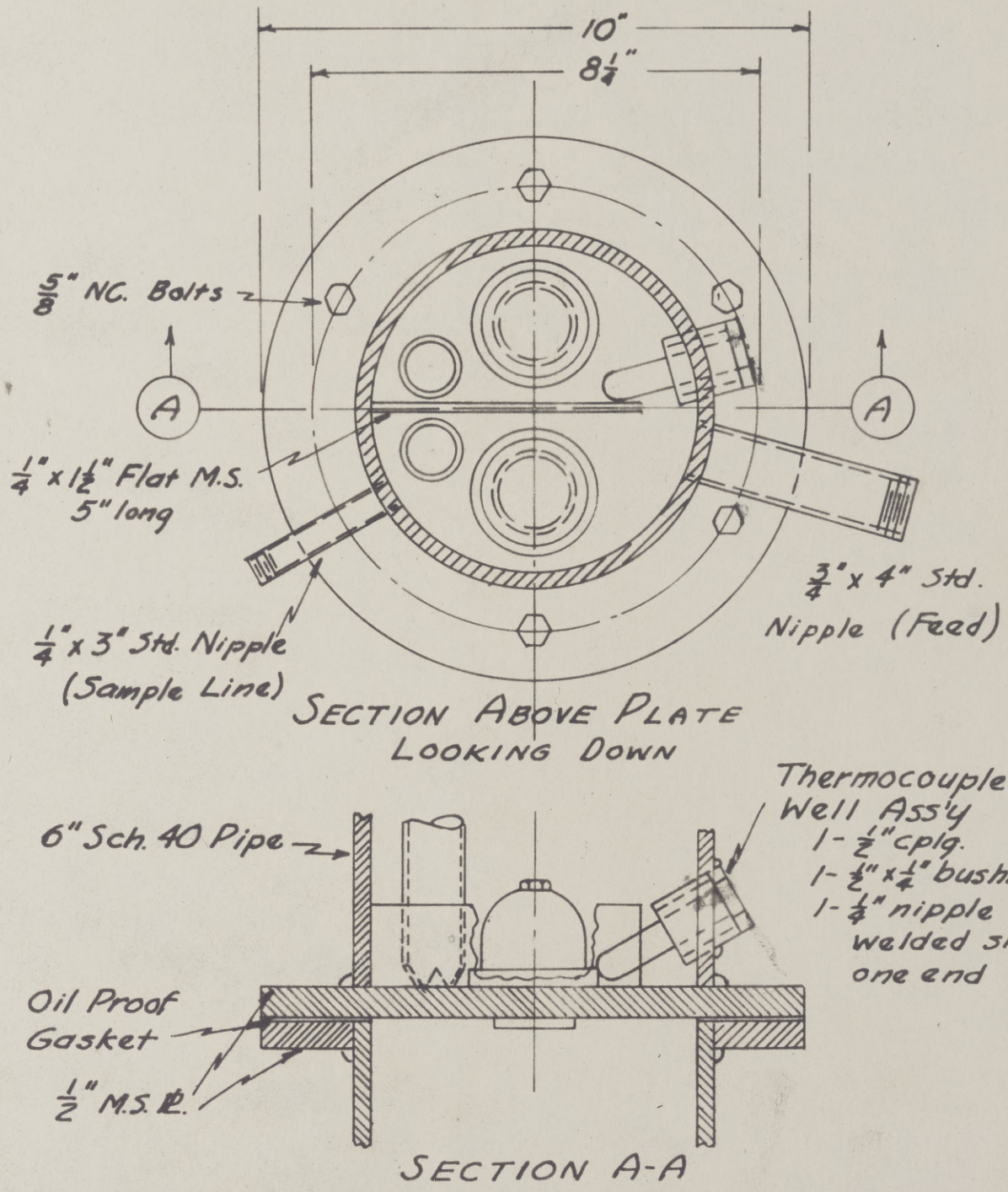
Fractionating Column

The column proper consists of flanged sections of 6-inch standard pipe, and has twelve plates, each spaced 12 inches apart. As seen on Figure 14, each plate has two $2\frac{1}{2}$-inch diameter Vulcan opposed-tangential-slot bronze bubble caps resting on the plate, and separated by a baffle so that the liquid passes each cap in series in travelling across the plate. Overflow pipes are one-inch standard pipe, and project through the plate to a height of one inch to form the overflow weir. The cap slots are 9/16" high, giving a minimum slot immersion of 7/16". The bottom end of each overflow pipe is notched out and rests on the plate below.

Plate 12 (the top plate) has an inlet for the reflux, and Plates 10, 8, 6, 4, and 2 have feed inlets, all of three-quarter inch pipe. Liquid sample lines from each plate are connected to individual coolers, consisting of coils of one-quarter inch copper tubing mounted in a common jacket of two-inch electrical conduit through which water flows.

Reboiler

This is made of a section of 18-inch outside-diameter line



Notes. 1. Bubble Caps are 2½" O.D. and have 18 slots 3/32" x ½"
2. Top of down pipe is 1" above plate

Fig. 14

PLATE ASSEMBLY DETAIL

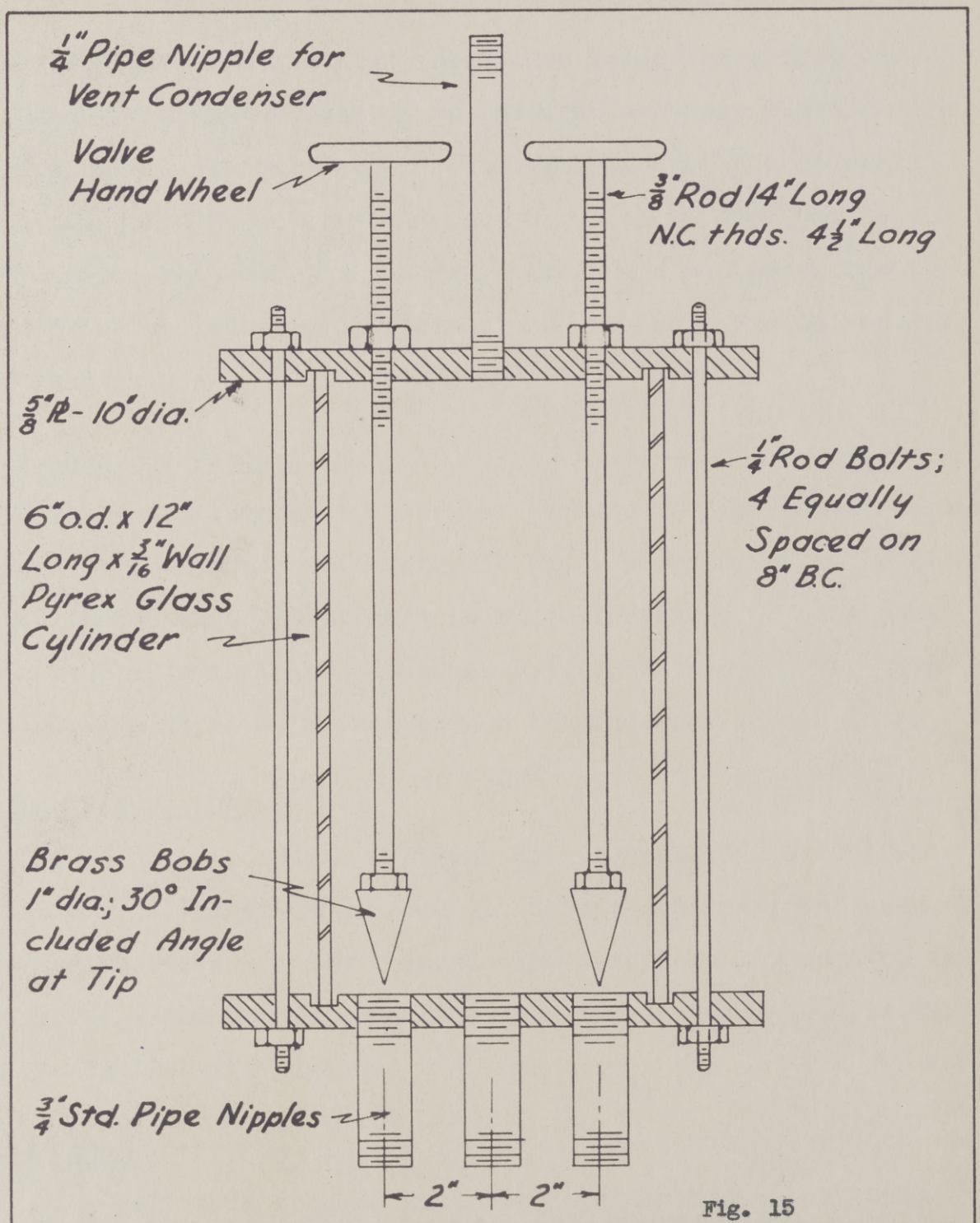


Fig. 15

REFLUX PROPORTIONER (CUT AWAY VIEW)

pipe 3 feet long and closed on one end by a welding cap. The other end has a welded flange to which is bolted a flat cover. The ends of the steam coil pass through the cover plate and the coil consists of twelve $2\frac{1}{2}$-foot lengths of one-inch standard pipe connected by return bends and arranged in tiers of 3, 4, and 5 pipes respectively. A gauge glass and manometer are included on the reboiler as well as the necessary drains, outlets, and inlet.

Condenser and Bottoms Cooler

These are identical pieces of equipment. Each is a double-pipe heat exchanger, with water in the annular space between the 2-inch and 3-inch pipes, made in three lengths of 5 feet each connected by ammonia-type condenser fittings and union return bends. The vapor pipe from column to condenser is a 2-inch.

Reflux Proportioner

This device, shown in Figure 15, receives all of the condensate from the condenser and divides it into overhead product and reflux streams. The tapered bobs were actually used to get coarse adjustment only, the fine adjustments being obtained with valves in the exit piping.

Insulation

The column is covered with standard moulded 85 per cent magnesia 6-inch pipe insulation. The protruding flanges are covered with a double layer of asbestos paper. Plastic magnesia is used to cover the reboiler, column top, and the tops and bot-

toms of the column plate flanges. All of this insulation is covered with Johns-Manville Insulkote. Vapor and steam piping and the feed line are lagged with standard magnesia pipe insulation.

Steam and Condensate Piping

All piping is standard. The pressure reducing valve on the steam line is a Klipfel ball-type valve, pilot-controlled and air-loaded, and gives very satisfactory control of steam pressure in the reboiler coil. A baffle-type condensate separator in the steam line immediately before the reboiler coil delivered approximately 99 per cent quality steam, as determined by a throttling calorimeter. Armstrong steam traps (inverted bucket type) are installed on both condensate separator and in the condensate line from the coil. The steam condensate can be either weighed in a collecting barrel or discharged to the drain.

Tanks

Three covered, welded, steel tanks are provided for receiving distillation products and for storage. These are equipped with gauge glasses and vent condensers and are so piped that any product can be put into any tank. The tanks are connected through a piping manifold to the inlet of a 50 gpm. Ingersoll-Rand centrifugal pump which delivers the materials to the feed tank (also of welded steel construction) which is located above the top of the column so that gravity feed can be used.

Flowmeters

Schutte and Koerting and Fischer and Porter rotameters in sizes from No. 1 to No. 4 are used to measure all hydrocarbon flows. Those with stainless steel floats were factory calibrated; those with duralumin (17 ST alloy) floats were calibrated in place. A No. 5 rotameter with stainless steel float measures the flow of water to the condenser. Orifice meters are installed on the condenser by-pass water line for measuring higher rates of flow than the rotameter will handle, and on the water line to the cooler.

Thermocouples

Iron-constantan single-junction thermocouples are used for all temperature measurements. These are provided with a 32-degree Fahrenheit cold junction by being led to oil-filled cold junction tubes which in turn are immersed in vacuum bottles containing water and ice. All couples are connected through a multi-point selector switch panel to a portable Leeds and Northrup potentiometer. Temperatures measured are: liquid on each plate, all streams entering and leaving both condenser and cooler, reboiler liquid, feed, steam condensate, and inlet and outlet of the steam calorimeter (see Table 14). Thermocouple wells are made of one-quarter inch pipe welded closed and filled with light lubricating oil. All thermocouples were calibrated in place, the resulting calibration curve giving a consistent but slightly low reading (constant negative difference of 0.11 millivolts) compared to standard tables. This enabled use of these tables merely by adding 0.11 millivolt to the observed EMF reading.

MATERIALS

Both the benzene and the toluene used in this work were "Industrial Pure" products donated by the Koppers Company, and presumably are from byproduct coke oven operations. The purification of these hydrocarbons has been completely described by Bowden,[4] and will therefore be only briefly outlined here.

Preliminary distillations on the original materials showed that both contained impurities boiling both higher and lower than the pure substances. In order to reduce the amounts of high-boiling impurities, the as-received hydrocarbons were rerun in the column and the bottoms rejected. The overhead products from these batch distillations were then azeotropically distilled in the column to remove the low-boiling impurities. Azeotrope-formers used were acetone for the benzene and methyl ethyl ketone for the toluene. Properties of both hydrocarbons, best cuts, and final products, are compared with accepted values for these constants in Table 5.

TABLE 5

PHYSICAL CONSTANTS OF HYDROCARBONS

Sample	n_D^{25}	d_4^{25}
A. Benzene		
Original material	1.49647	0.8728
Purified - Cut 40	1.49686	0.8730
Purified - Cut 41	1.49701	0.8732
Purified - composite	1.49628	-
Literature - Ref. 31	1.4979	0.8737
B. Toluene		
Purified - Cut 22	1.49338	0.8621
Purified - composite	1.49328	-
Literature - Ref. 31	1.4940	0.8621

PROCEDURES AND CALCULATIONS

Analytical Method

The method used to analyze the mixtures of benzene and toluene column samples was the boiling point of the liquid. Boiling-point apparatus was constructed in duplicate and was the modified Cottrell boiling point apparatus described by Griswold and coworkers,[17] further modified by the addition of a drain tube with stopcock extending out from the bottom of the tube and sloped slightly downwards from the horizontal to facilitate draining of a used sample.

Known mixtures were run in each apparatus to obtain an overall boiling point-composition calibration curve for these instruments. The original data for this chart and the quantities calculated from it are given in Table 6 and in graphical form in Figure 16.

The corrections to observed boiling point for deviation of barometric pressure from 760 mm. were made throughout by use of an average value of (dt/dp). Since the difference in (dt/dp) for benzene and for toluene is small, the use of an average was considered justified. Values of (dt/dp) given in the tables[33] are from API Research Project No. 41.

Runs to Determine Heat Loss

Fourteen runs were made to determine the heat loss from the column and other parts of the equipment. All of these runs were made at total reflux in order to simplify calculation. After operating the equipment for some hours to attain thermal

TABLE 6

BOILING POINT CALIBRATION CURVE

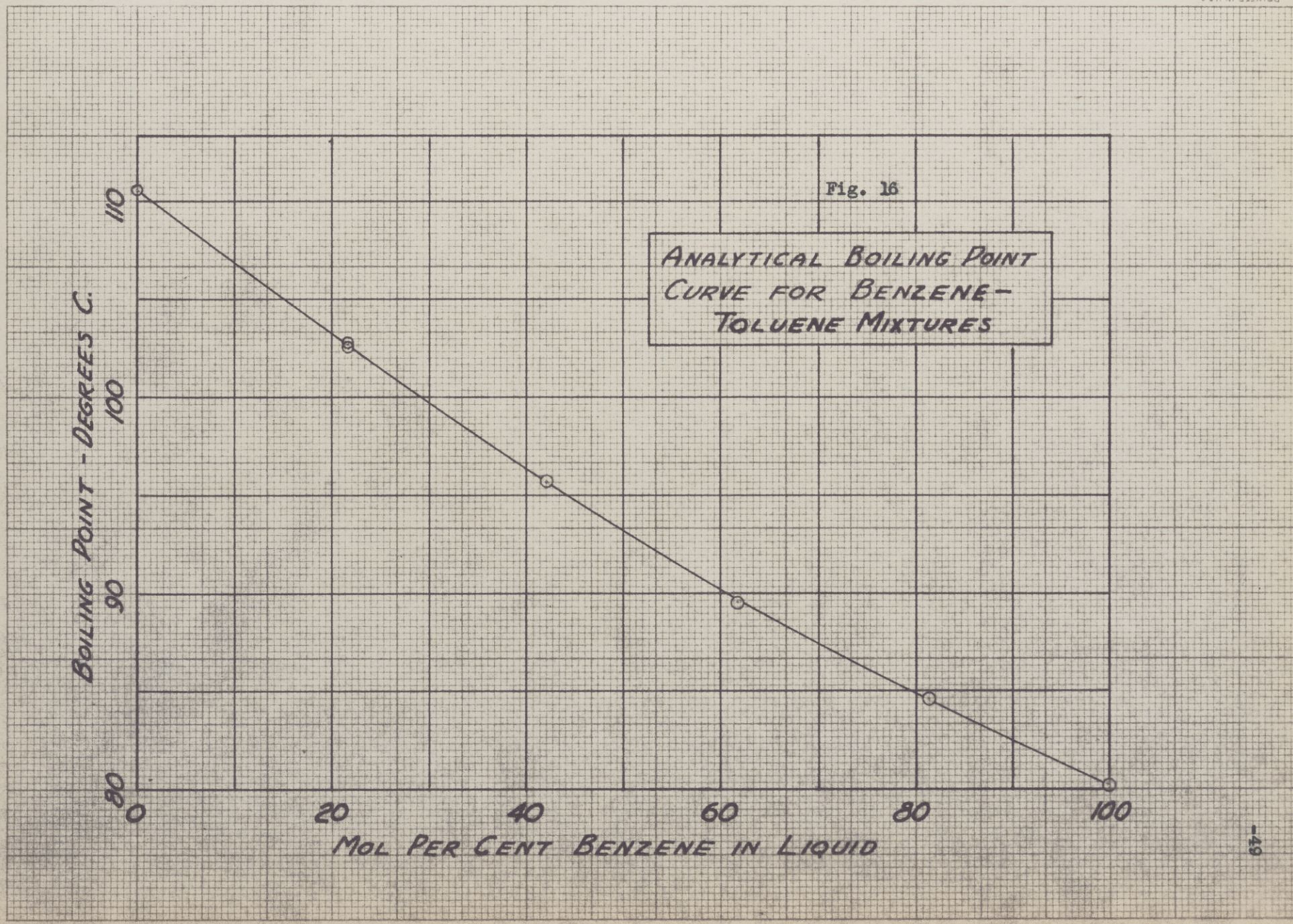
Solution mol % C_6	Apparatus Number	Obs. B.P. °C.	Barom. mm. Hg.	B.P. 760 mm.
0	1	110.1	751.5	110.5
	2	110.1	751.5	110.5
21.15	1	102.3	752.0	102.7
	2	102.4	751.5	102.8
42.1	1	95.3	751.5	95.7
	2	95.4	751.5	95.8
61.7	1	89.2	751.5	89.6
	2	89.3	751.5	89.7
81.2	1	84.2	751.5	84.6
	2	84.1	751.5	84.5
100	1	79.8	752	80.2
	2	79.8	752	80.2

equilibrium, the following data were taken: weight of steam condensate collected during a measured time interval, steam pressure, steam condensate temperature, water rate to the condenser, and inlet and outlet temperatures of the condenser water.

On one run, surface temperatures of various parts of the equipment were measured, and these, with known areas and calculated heat transfer coefficients, enabled an approximation of heat loss from each component part, so that the total could be correctly apportioned among the various parts of the unit.

Summaries of the data on these runs and of the calculations made from them are given in tables in the Appendix.

Fig. 16



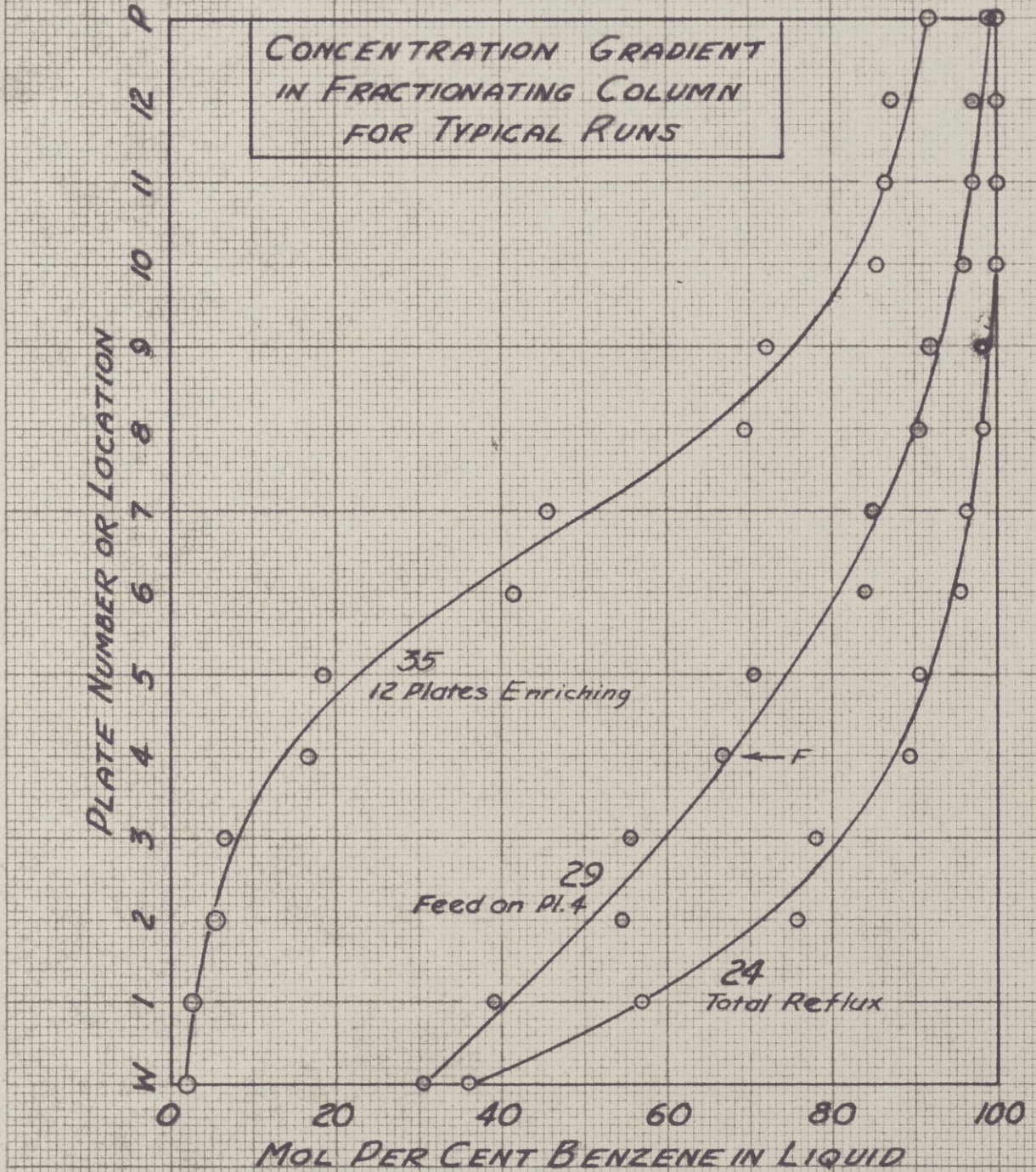
Total Reflux Runs

These runs were made in several series. Here the only variables of interest are vapor velocity and composition of the liquid. Ordinarily some 8 to 10 hours (much longer than was necessary as proved by subsequent partial reflux runs) was allowed for the attainment of equilibrium. The sample lines were purged about an hour before taking samples of the liquids. At the end of the run, temperatures of all plates, the reboiler, and the condenser streams, and all flow rates were recorded. Then liquid samples of about 25 ml. were taken as fast as possible of overhead product, from all plates, and from the reboiler.

The conclusion of one run was frequently the beginning of another, for it was only necessary to change the setting on the steam pressure regulating valve to alter the vapor velocity. From there on the entire process was repeated.

From plots of plate liquid composition against plate location (such as Figure 17) it was apparent that the sample outlets were not located at the same point on all plates. It will be noted on Figure 17 that many of the points tend to "pair up"; this is explained on the basis that on one plate the sample line is relatively close to where the liquid leaves the plate, and that on the plate below the sample line is in a symmetrical location with respect to where the liquid is discharged onto the plate. Accordingly, the average composition of the liquid on any plate was taken to be that given by the curve drawn through the data when plotted as in Figure 17. This applies both to total and to partial reflux runs. The existence of marked concentration

Fig. 17



gradients in the liquid on the plates of a small column has been conclusively demonstrated.[15]

The calculation of overall plate efficiency was made by the McCabe-Thiele graphical method. For runs in which the liquid composition in the upper part of the column approached pure benzene, slight analytical inaccuracies are greatly magnified in plate calculations and the starting point for the computation of the number of theoretical plates was chosen as that plate on which the liquid was approximately 95 per cent benzene. The reboiler was assumed to be equivalent to one theoretical plate, and accordingly, one theoretical plate was deducted from the total so as not to include the effect of the reboiler. A specimen McCabe-Thiele plot is given as Figure 18.

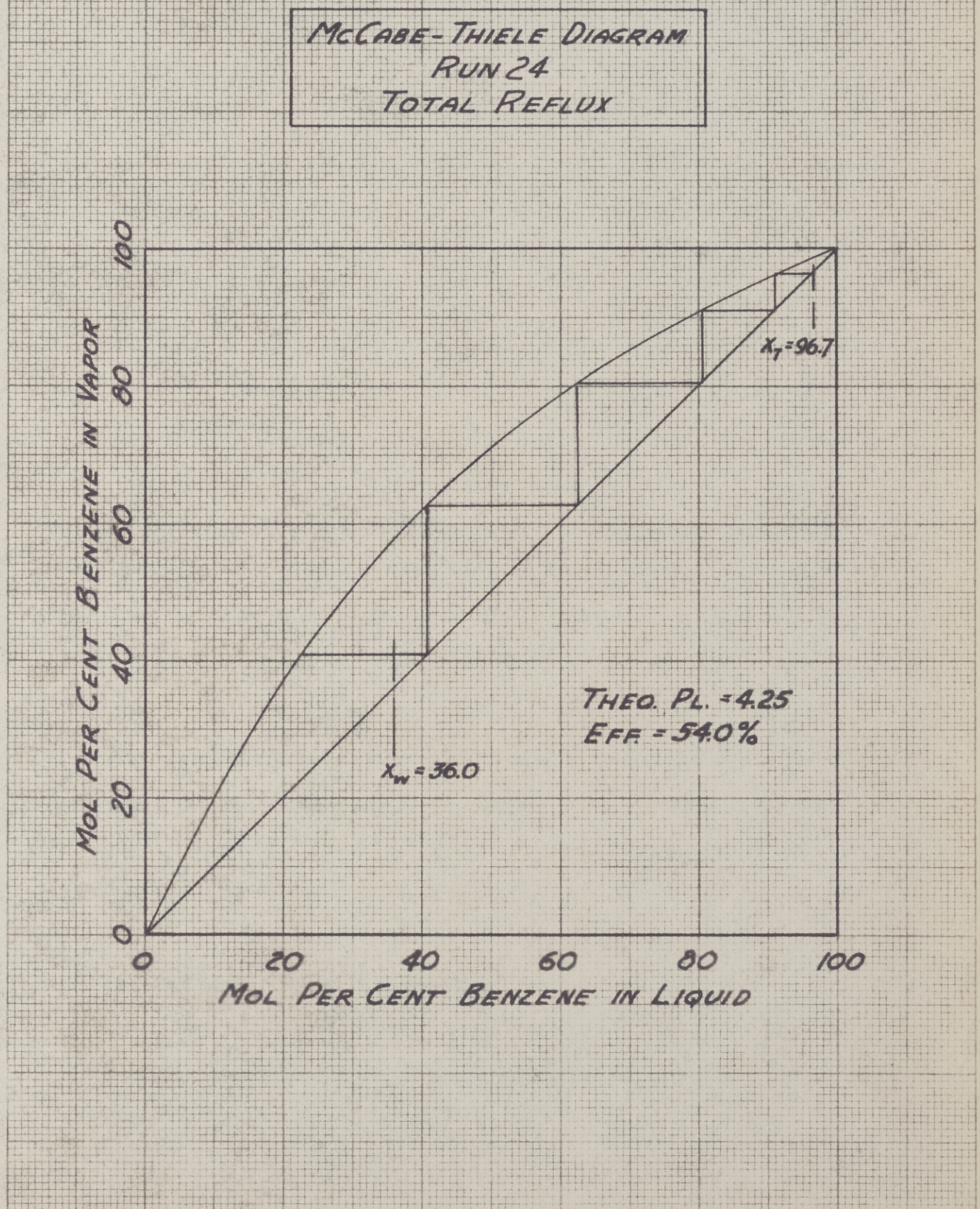
A summary of the original data, of the results calculated from them, and sample calculations are given in the Appendix.

Partial Reflux Runs

These runs can be subclassified into: (1) those in which the feed was introduced on one of the plates in the column, and (2) those in which the overhead product was recycled to the reboiler (12 plate enriching column).

The unit was started up at total reflux for all runs and left on total reflux until temperatures became constant. Then the feed valve was opened and adjusted to the desired position, and the overhead and bottoms products streams started. There was seldom sufficient feed stock on hand to run long enough to insure steady conditions; consequently the products streams were mixed and recycled to the feed tank at intervals.

Fig. 18



When plate temperatures remained essentially constant for about an hour or more, it was assumed that equilibrium had been attained, and samples were drawn, using the same procedure as in total reflux runs.

Equivalent theoretical plates for partial reflux runs were also calculated by the McCabe-Thiele method. The two terminal compositions, overhead and bottoms compositions, were available from the sample analyses. The slope of the enriching line was calculated from the metered flows of reflux and of overhead product, corrected for vapor condensed by the cold reflux and the heat loss from the column. Both of these corrections were made by calculating (see sample calculation for Run 29 in Appendix) the vapor condensed and adding this quantity to both liquid and vapor streams. For the heat loss correction, an average heat loss of 5 Btu. per minute for each column section of one plate and an average latent heat were used. The heat loss correction was significant only at vapor rates of about 0.5 feet per second and less.

The intersection of the operating lines was taken as the experimental feed plate liquid composition. From this intersection a straight line was drawn to the intersection of the x coordinate of the bottoms composition and the 45 degree line. Then the number of theoretical plates was stepped off in the customary manner, as illustrated in Figure 19.

The calculation for the twelve-plates-enriching column operation was also made by the McCabe-Thiele method. Here the

Fig. 19

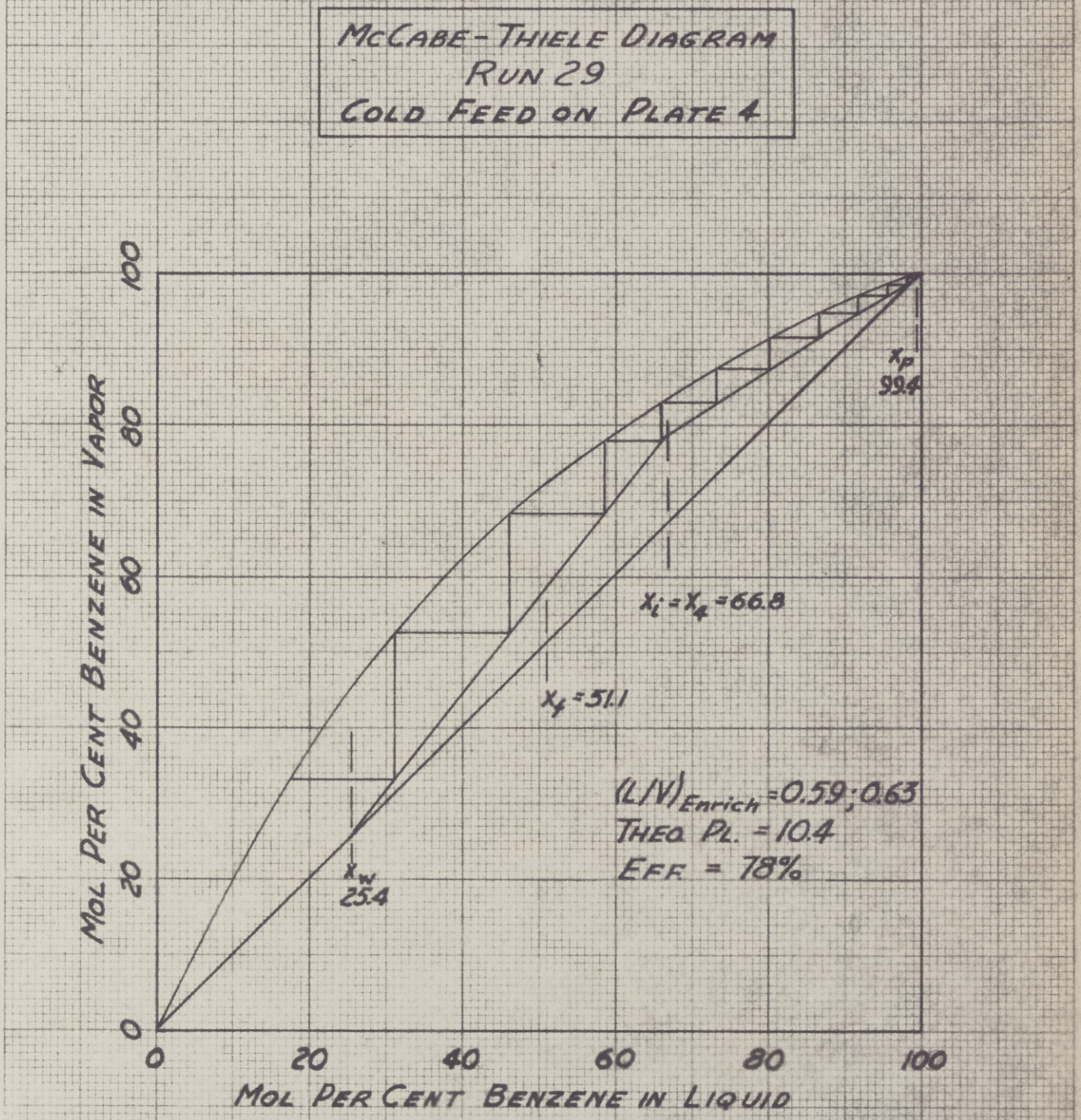
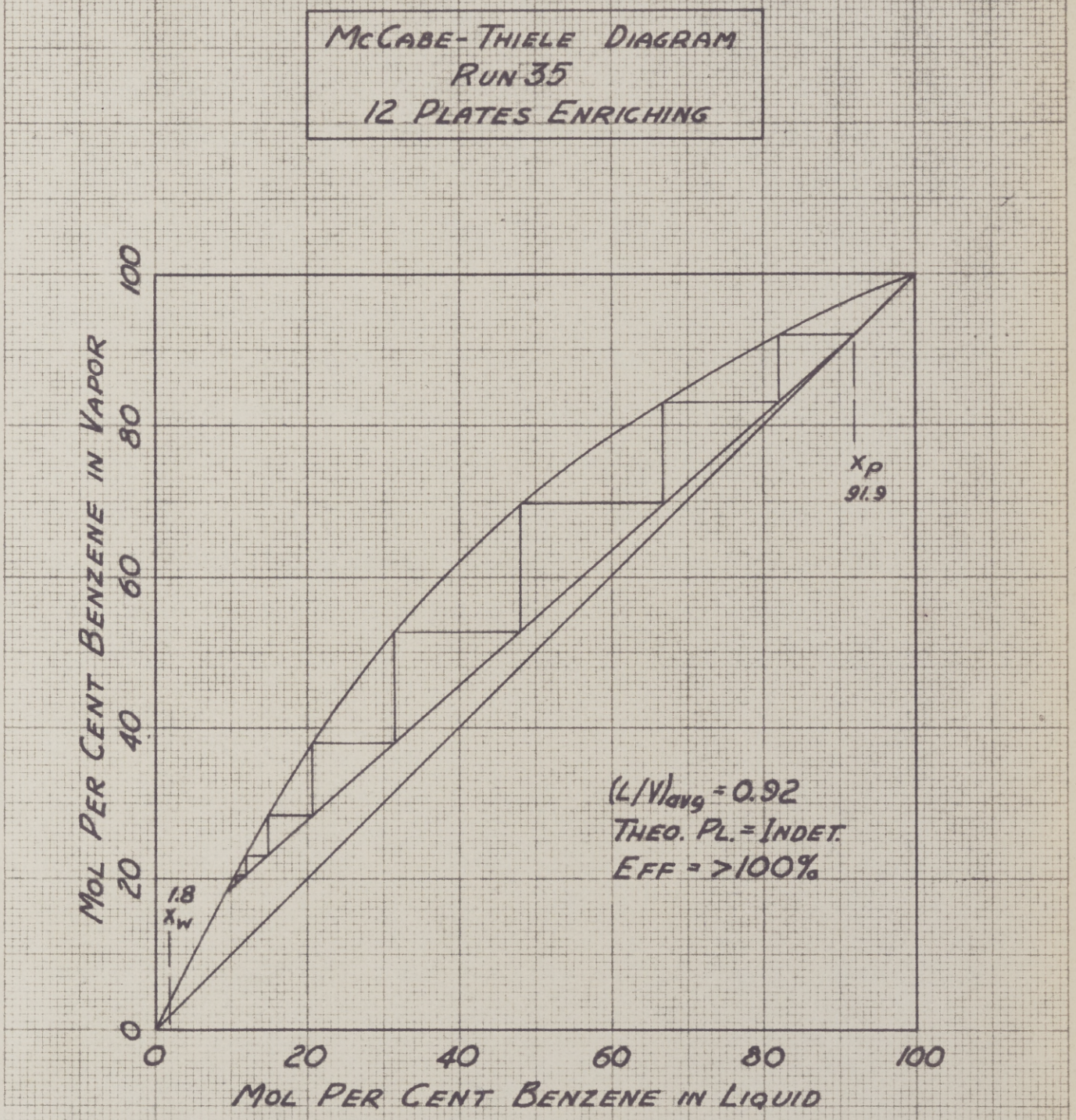


Fig. 20



one operating line was determined by the overhead composition and the slope calculated from the metered overhead quantities and corrected for cold reflux and heat loss at several points. This is illustrated in Figure 20.

Summaries of the original data for all partial reflux runs are given in the Appendix, as well as sample calculations and a tabular summary of the various calculated quantities.

Murphree Plate Efficiencies

The Murphree plate efficiencies for several of the runs were calculated for comparison with the overall efficiencies, and to determine the effect of concentration on efficiency. Figure 21 shows the graphical method of determining the corresponding values of y and y^* from plate liquid samples needed to calculate the Murphree individual plate efficiency. The formula for this is given by Griswold[16] and by Underwood[55] for "plate (n)" as

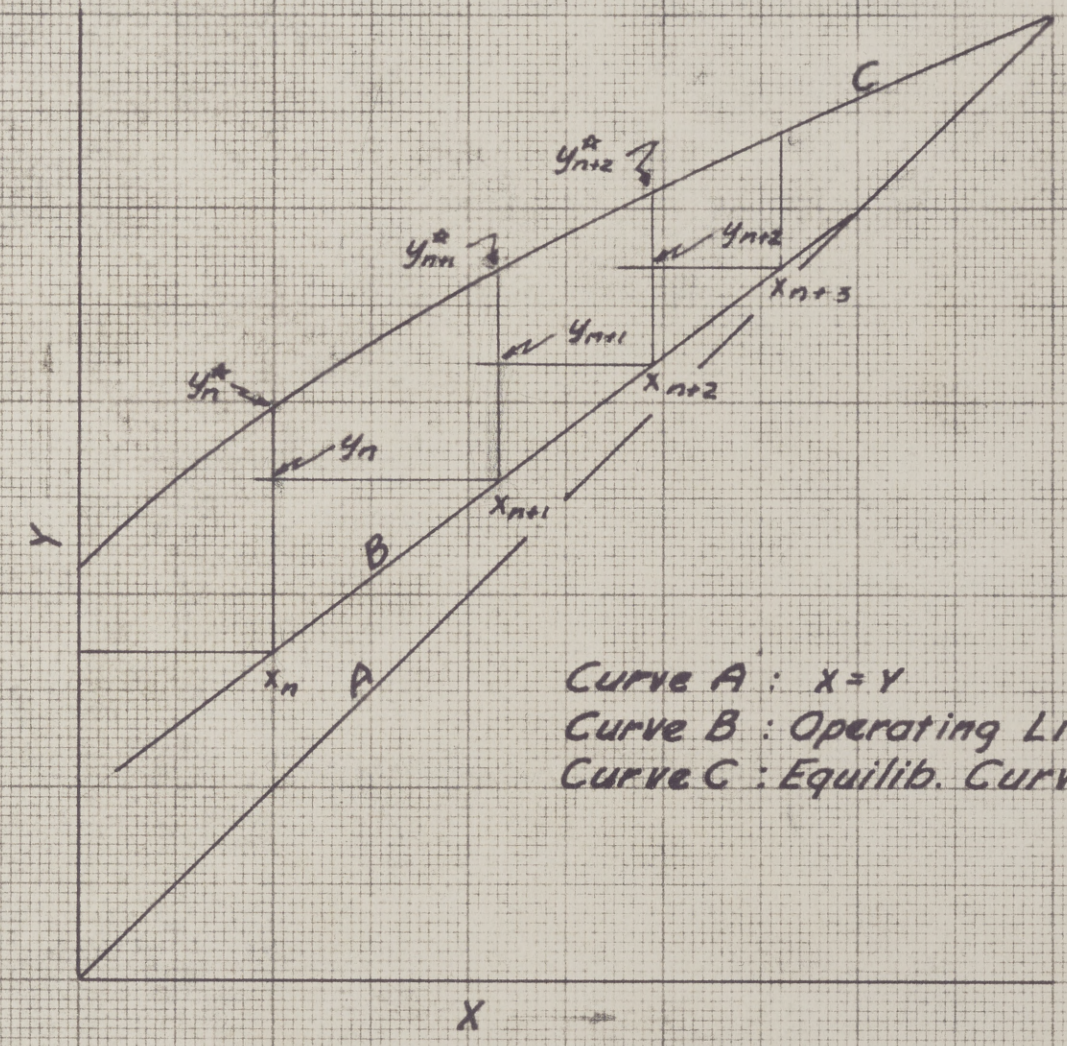
$$\eta = \eta_x = \eta_y = \frac{x_n - x_{n-1}}{x_n^* - x_{n-1}} = \frac{y_n - y_{n-1}}{y_n^* - y_{n-1}}$$

where η_x equals efficiency calculated for liquid compositions and x_n^* is the composition of liquid in equilibrium with y_n.

Tabular calculations are given in the Appendix.

Fig. 21

METHOD FOR GRAPHICAL DETERMINATION FROM PLATE LIQUID SAMPLES OF OTHER QUANTITIES IN MURPHREE EFFICIENCIES



V

APPENDIX

RUNS TO DETERMINE HEAT LOSS

Table 7	Original Data and Calculations

TOTAL REFLUX RUNS

Table 8	Summary of Original Data
Table 9	Calculations Summary

PARTIAL REFLUX RUNS

Table 10	Summary of Original Data
	Sample Calculations
Table 11	Calculations Summary

INDIVIDUAL PLATE EFFICIENCIES

Table 12	Murphree Efficiency Calculations
Table 13	Recalculation of Murphree Efficiency to Liquid Film Basis

THERMOCOUPLE INSTALLATION

Table 14	Thermocouple Installation

TABLE 7
RUNS TO DETERMINE HEAT LOSS

A ORIGINAL DATA

Run No.	6A	6B	7A	7B	8A	8B	9A	9B	10A	10B	10C	11A	11B	11C
Cond. H_2O out; t - °F.	107	88	103	99	118	110	111	108	112	110	107	120	130	141
Cond. H_2O in; t - °F.	84	84	87	86	86	86	84	86	88	89	90	84	86	86
Cond. H_2O rate; lb/min.	27.9	27.2	27.5	18.6	17.75	17.6	18.75	17.4	16.0	16.4	11.2	4.4	4.2	4.2
Steam Cond; lb/min.	0.85	0.267	0.60	0.35	0.70	0.516	0.718	0.525	0.75	0.528	0.367	0.300	0.345	0.900
Steam Press.; psig	26	15.5	20	15.25	25	20	24.5	20.5	26	20.5	16.5	5.33	6.33	7.33
Steam Cond. t. - °F.	269	251	260	251	267	259	266	259	263	257	249	226	230	232
Room temp. - °C.	36	36	36	35	36	35	35.5	35.5	35	35.5	34.5	25	25.5	25.5

B CALCULATIONS

Cond. H_2O - Δt - °F.	23	4	16	13	32	24	29	22	24	21	17	36	44	55
H, steam - Btu/lb.	1160	1155	1158	1154	1160	1158	1160	1158	1159	1157	1152	1155	1157	1158
H, cond. - Btu./lb.	238	219	228	220	236	228	234.5	227.5	231	222.5	217.5	194	198	201
Heat in - Steam Btu/min.	985	308	695	404	812	598	833	608	869	610	422	347	399	463
Heat out - Cond. Btu/min.	202	58	137	77	165	118	168	119	173	119	79	59	68	81
Heat out - H_2O - Btu/min.	642	109	440	242	568	422	544	383	544	345	191	158	185	231
Heat out - Total - Btu/min.	844	167	577	319	733	540	712	502	717	464	270	217	253	312
HEAT LOSS - Btu/min.	141	141	118	85	79	58	121	106	152	146	152	130	146	151

TABLE 8 ORIGINAL DATA - TOTAL REFLUX RUNS

RUN No.		1			2			3			4			5			6			7			8			9			10			11			12			14			22			24		
		Temp °F	Liq. Comp: Mol % O Samp.	Corrd.	Temp °F	Liq. Comp: Mol % O Samp.	Corrd.	Temp °F	Liq. Comp. Mol % O Samp.	Corrd.	Temp °F	Liq. Comp. Mol % O Samp.	Corrd.	Temp °F	Liq. Comp. Mol % O Samp.	Corrd.	Temp °F	Liq. Comp. Mol % O Samp.	Corrd.	Temp °F	Liq. Mol Samp	Comp % O Corrd	Temp °F	Liq. Comp. Mol % O Samp.	Corrd.	Temp °F	Liq. Comp. Mol % O Samp.	Corrd.	Temp °F	Liq. Comp. Mol % O Samp.	Corrd.	Temp °F	Liq. Comp. Mol % O Samp.	Corrd.	Temp °F	Liq. Comp. Mol % O Samp.	Corrd.	Temp °F	Liq. Comp. Mol % O Samp.	Corrd.	Temp °F	Liq. Comp. Mol % O Samp.	Corrd.
Column - Vapor out		179	94.1	94.1	176	100	100	177	100	100	172	100	100	176	100	100	179	100	100	178	100	100	176	100	100	176	100	100	175	100	100	178	100	100	177	100	100	178	100	100	176	100	100	175	100	100
- Plate	12	179	91.0	91.2	176	100	100	177	100	100	173	100	100	176	100	100	179	96.7	98.2	178	100	100	176	100	100	176	100	100	175	100	100	178	100	100	177	100	100	179	99.4	99.7	177	100	100	176	100	100
	11	181	88.4	88.5	177	100	100	178	100	100	174	100	100	177	100	100	179	96.2	95.8	178	100	100	177	100	100	177	100	100	176	100	100	178	100	100	177	100	100	-	99.4	99.2	178	100	100	178	100	100
	10	181	85.9	85.0	177	100	99.0	177	100	100	174	100	100	176	100	100	180	95.1	92.8	178	100	100	176	100	100	177	100	100	175	100	100	178	100	100	177	100	100	-	98.3	98.4	178	99.4	99.2	177	98.2	99.2
	9	186	82.2	81.9	179	99.4	99.2	179	99.4	99.2	176	98.4	99.2	177	98.3	98.7	184	85.5	88.8	179	96.2	96.8	178	99.4	99.3	178	97.2	98.5	175	97.7	98.5	179	97.7	98.8	178	99.4	99.5	178	97.7	97.3	179	95.6	94.8	178	96.7	97.0	177	98.2	99.2
	8	186	82.2	78.4	179	96.2	96.0	178	98.8	98.1	176	97.7	97.4	176	97.7	96.7	185	83.0	84.3	179	94.6	93.0	179	92.2	93.0	179	96.7	96.7	175	97.2	96.4	180	97.2	97.5	179	98.3	98.1	180	95.1	95.8	178	96.2	96.7	179	90.1	91.5	178	98.2	98.2
	7	192	73.1	74.5	181	92.3	92.8	181	97.0	96.5	178	92.3	94.4	178	92.7	94.1	191	79.1	79.1	180	88.4	88.5	178	85.6	86.2	178	92.1	93.8	174	92.7	93.7	179	97.2	95.4	179	98.3	98.1	181	97.2	96.3	183	88.8	85.8	179	95.6	94.8	183	88.8	85.8
	6	196	69.5	69.8	184	91.0	87.4	182	93.8	93.7	179	91.0	90.0	180	91.5	90.0	195	75.8	73.7	187	79.5	82.8	181	94.1	93.5	182	91.5	89.3	173	91.9	90.2	180	95.1	92.0	181	97.2	96.3	181	94.6	93.6	185	75.4	77.5	179	95.6	94.8	185	75.4	77.5
	5	200	61.1	64.0	189	72.4	73.5	183	87.0	88.5	182	81.0	84.0	182	81.4	84.0	198	63.8	67.0	193	72.1	75.0	184	89.2	89.3	185	82.2	83.2	179	83.0	85.0	181	88.0	87.0	182	95.1	95.4	181	88.8	90.9	189	72.1	67.5	182	89.2	87.3
	4	205	57.8	56.3	194	69.5	70.2	187	80.7	81.2	185	79.1	76.0	185	79.5	75.8	202	62.5	59.7	197	69.2	67.7	186	85.9	84.3	189	77.3	75.5	185	79.1	76.5	184	75.4	80.0	183	93.2	93.2	186	85.1	86.4	190	50.1	55.5	183	78.0	81.0
	3	211	44.4	46.4	199	58.1	60.3	191	69.2	72.0	191	59.2	65.8	191	59.3	65.5	209	47.5	51.1	200	57.1	60.0	189	73.5	75.0	192	61.5	65.6	190	58.1	65.2	190	65.4	69.7	183	89.4	90.0	187	75.8	79.4	203	47.5	42.0	189	75.8	71.7
	2	216	39.8	35.5	204	55.4	49.1	196	67.0	60.9	200	59.2	54.3	198	55.4	52.5	214	46.6	41.6	207	51.5	51.5	197	66.1	61.2	200	62.2	52.5	199	54.1	50.8	199	55.4	53.2	186	86.7	83.1	193	74.3	68.8	211	25.1	27.0	194	57.0	56.3
	1	226	27.5	23.8	213	39.1	38.2	205	43.8	43.0	208	31.5	41.2	208	30.6	36.9	223	31.5	30.0	215	37.6	39.5	208	39.5	41.2	209	33.1	35.5	208	27.5	34.0	209	29.3	34.2	193	69.2	69.5	200	50.4	51.2	215	11.3	11.3	203	35.9	36.0
Reboiler		-	11.5	11.5	-	21.7	21.5	-	37.8	34.0	-	37.3	27.5	-	31.8	19.5	-	9.2	9.2	-	14.3	14.3	-	14.6	14.6	212	15.7	15.7	210	15.1	15.0	216	12.0	12.0	204	41.6	41.5	206	28.7	28.1	215	11.3	11.3	203	35.9	36.0
Condenser - H_2O in		85			84			86			84			87			88			86			87			86			81			90			84						87			89		
- H_2O out		128			123			118			109			116			127			125			123			113			85			106			120						115			125		
- Prod. out		137			133			123			100			93			134			135			134			112			83			91			127						124			137		
Control Air Press. - psig		26.5			21.75			17.38			13.5			10.5			26.25			21.15			17.25			13.5			10.9			9.5			10.75						14.4			-		
Steam Press. - psig		25			20.25			15.5			11.5			9.0			25			20			15.5			11.75			9.0			7.5			8.75						13.5			-		
Reflux - gpm		0.601			0.56			0.475			0.285			0.155			0.60			0.57			0.635			0.40			0.035			0.03			0.495						0.225			0.305		
Room Temp. - °C.		-			29			29			28			29			31			31			31			31			29			29			-						-			-		
Barometer - mm Hg.		-			743			744			745			746			745			744			746			747			746			746			-						-			-		

TABLE 9

CALCULATIONS SUMMARY
TOTAL REFLUX RUNS

RUN No.	Vapor Velocity ft per sec.	Overall Efficiency	Mol % O top Plate used in calc.	Mol % O Reboiler	Theoretical Plates	Actual Plates	Avg. Plate Comp. Mol % O	Diff. Avg. Plate Comp. from 65%	Concn. Correction	Efficiency Corrected to 65% Avg. Conc.
1	1.96	36.4	94.1	11.5	5.37-1	12	66.2	1.2	0.7	37.1
6	1.96	48.9	95.8	9.2	5.89-1	10	71.1	6.1	3.7	52.6
7	1.87	54.3	93.0	14.3	4.80-1	7	69.8	4.8	2.9	57.2
2	1.83	55.9	96.0	21.5	4.91-1	7	71.8	6.8	4.1	60.0
8	1.75	72.0	96.2	14.6	5.32-1	6	77.3	11.3	6.8	78.8
12	1.62	58.2	96.9	41.5	3.91-1	5	88.0	23.0	13.8	72.0
14	1.62	54.8	95.8	28.7	4.29-1	6	80.9	15.9	9.6	64.4
3	1.55	53.3	96.5	34.0	4.20-1	6	77.4	12.4	7.5	60.8
9	1.31	62.2	93.8	15.7	4.73-1	6	70.8	5.8	3.5	65.7
24	1.01	51.0	96.7	36.0	4.06-1	6	82.8	17.8	10.7	61.7
4	0.93	55.0	97.4	27.5	4.85-1	7	75.4	10.4	6.3	61.3
22	0.72	63.7	94.8	11.3	5.46-1	7	67.8	2.8	1.7	65.4
5	0.51	58.6	96.7	19.5	5.10-1	7	74.4	9.4	5.6	64.2
10	0.31	62.9	96.4	15.0	5.40-1	7	74.0	9.0	5.4	68.3
11	0.098	75.5	95.4	12.0	5.53-1	6	73.2	8.2	4.9	80.4

TABLE 10 ORIGINAL DATA – PARTIAL REFLUX RUNS

Run No.		15			16			17			18			19			20			21			23			25		
		Temp °F	Liq. Comp. Mol % O Samp.	Liq. Comp. Mol % O Corrd	Temp °F	Liq. Comp. Mol % O Samp.	Liq. Comp. Mol % O Corrd	Temp °F	Liq. Comp. Mol % O Samp.	Liq. Comp. Mol % O Corrd	Temp °F	Liq. Comp. Mol % O Samp.	Liq. Comp. Mol % O Corrd	Temp °F	Liq. Comp. Mol % O Samp.	Liq. Comp. Mol % O Corrd	Temp °F	Liq. Comp. Mol % O Samp.	Liq. Comp. Mol % O Corrd	Temp °F	Liq. Comp. Mol % O Samp.	Liq. Comp. Mol % O Corrd	Temp °F	Liq. Comp. Mol % O Samp.	Liq. Comp. Mol % O Corrd	Temp °F	Liq. Comp. Mol % O Samp.	Liq. Comp. Mol % O Corrd
Column - Vapor out		180	95.1	95.0	178	97.1	97.1	178	98.8	98.8	179	95.6	95.6	181	95.1	95.0	181	94.2	94.2	182	94.6	94.6	177	90.8	90.8	179	96.2	96.2
- Plate	12	180	88.8	90.0	178	93.2	94.4	178	93.6	91.0	178	88.8	90.3	180	87.6	90.2	180	86.7	89.2	181	85.9	87.0	177	94.1	96.1	179	90.1	92.6
	11	180	86.1	84.8	178	92.1	91.7	179	95.6	95.3	179	88.4	85.8	181	85.1	84.6	182	84.2	83.3	183	83.0	80.7	178	93.2	92.8	180	89.2	88.8
	10	180	81.4	78.1	178	90.5	89.4	179	94.6	93.7	179	84.7	81.0	–	79.5	77.0	–	78.1	75.1	–	79.1	75.0	–	90.5	88.9	–	85.9	84.5
	9	185	76.3	76.0	180	85.1	82.4	181	90.1	91.8	184	77.7	78.3	187	74.3	75.7	185	72.4	74.4	190	65.8	69.4	181	84.2	84.3	184	76.9	79.1
	8	186	74.6	74.7	181	85.1	85.0	183	88.8	89.4	184	78.4	77.7	187	74.3	74.2	187	73.1	72.8	194	61.8	64.1	182	80.7	79.5	186	70.3	70.3
	7	186	72.4	73.0	182	81.0	82.0	183	88.4	86.2	184	76.5	76.3	187	72.1	72.1	187	69.9	70.8	193	53.4	58.8	184	79.5	74.5	188	67.8	68.6
	6	188	72.1	70.6	185	80.7	78.5	181	82.6	81.7	186	75.0	74.4	190	71.7	69.7	191	62.5	68.0	196	53.4	53.5	187	69.5	69.5	191	66.4	66.8
	5	191	65.8	67.3	187	72.5	74.0	189	74.3	75.7	189	70.3	71.6	192	64.2	66.0	193	61.5	63.3	205	41.9	43.5	191	64.6	65.6	182	64.6	64.6
	4	193	64.2	61.8	191	70.3	67.9	194	69.9	68.5	191	67.8	67.5	195	62.2	60.7	197	58.8	56.8	212	38.8	33.6	192	64.2	63.5	194	63.2	61.6
	3	195	51.5	54.4	193	53.1	58.4	197	56.1	59.5	194	57.1	60.0	198	49.1	53.2	193	47.2	48.1	218	18.1	24.3	193	58.8	59.7	195	55.4	52.8
	2	200	50.4	44.0	202	55.4	45.5	206	51.5	46.4	201	53.1	43.5	204	47.5	43.0	207	43.2	38.4	224	17.6	16.0	196	53.1	54.4	199	54.7	51.1
	1	208	31.2	31.4	210	29.0	30.5	213	30.3	36.3	207	35.6	34.5	210	36.3	29.6	213	24.5	26.0	228	6.8	9.3	199	46.3	46.0	204	40.1	40.5
Reboiler		208	17.9	17.5	213	14.9	15.0	211	13.5	13.5	209	16.5	16.5	211	15.1	15.0	210	13.5	13.5	225	7.6	4.0	197	33.4	33.5	209	24.8	24.5
Condenser - H_2O in		90			86			92			88			87			86			89			86			88		
- H_2O out		118			105			115			103			100			100			133			108			113		
- Prod. out		94			99			119			93			91			90			142			108			113		
Feed		89	57.1		93	63.9		93	69.2		92	66.4		88	61.9		92	63.5		91	67.4		90	57.1		94	57.4	
Control Air Press. psig.		10.5			14.25			19.25			10			11.8			14.5			17.75			4.3			7.75		
Steam Press. - psig.		9			12.5			18			9.5			10.5			12.5			15.5			2.5			6.25		
Reflux	gpm.	0.10			0.265			0.42			0.10			0.08			0.08			0.09			0.08			0.09		
Product	gpm	0.039			0.040			0.040			0.052			0.052			0.052			0.064			0.054			0.062		
Feed	gpm	0.077			0.073			0.075			0.16			0.10			0.10			0.11			0.108			0.85 #/min.		
ΔP across col.	"Hg.	1			1.4			2.2			0.9			1			1			1			0.85			0.9		
Feed Plate		10			10			10			10			10			10			6			6			8		

Run No.		26			27			28			29			32			33			34			35			36		
		Temp °F	Liq. Comp. Mol % O Samp.	Liq. Comp. Mol % O Corrd	Temp °F	Liq. Comp. Mol % O Samp.	Liq. Comp. Mol % O Corrd	Temp °F	Liq. Comp. Mol % O Samp.	Liq. Comp. Mol % O Corrd	Temp °F	Liq. Comp. Mol % O Samp.	Liq. Comp. Mol % O Corrd	Temp °F	Liq. Comp. Mol % O Samp.	Liq. Comp. Mol % O Corrd	Temp °F	Liq. Comp. Mol % O Samp.	Liq. Comp. Mol % O Corrd	Temp °F	Liq. Comp. Mol % O Samp.	Liq. Comp. Mol % O Corrd	Temp °F	Liq. Comp. Mol % O Samp.	Liq. Comp. Mol % O Corrd	Temp °F	Liq. Comp. Mol % O Samp.	Liq. Comp. Mol % O Corrd
Column - Vapor out		179.5	97.1	97.1	179	98.8	98.8	178	98.4	98.4	178	98.4	98.4	181	92.3	92.3	181	92.7	92.1	184	84.2	84.2	183	91.9	91.9	188	83.4	83.5
- Plate	12	179.5	94.1	96.5	179	97.2	97.1	178	96.7	97.8	179	96.1	96.1	–	83.8	81.7	–	85.1	89.0	–	71.3	77.7	–	87.2	89.5	–	78.0	82.4
	11	180	92.7	93.8	180	96.7	96.5	179	96.1	96.1	179	97.2	96.7	182	82.8	82.5	182	83.8	84.0	189	69.9	69.3	183	84.7	86.7	188	80.7	81.0
	10	–	90.5	90.0	–	95.1	95.0	–	94.6	94.0	180	96.1	95.0	–	79.5	76.6	–	81.8	78.4	–	64.4	61.2	–	85.5	82.2	–	80.7	79.0
	9	181	84.2	85.0	181	92.3	92.9	180	91.0	91.5	180	91.3	92.7	189	64.6	63.3	188	68.5	71.5	198	49.1	52.0	188	72.4	75.6	201	73.9	75.7
	8	185	80.7	79.4	183	91.2	90.1	181	87.6	88.5	181	90.5	89.9	193	62.5	61.0	190	65.0	65.0	203	46.6	44.0	194	63.6	65.0	209	70.9	71.8
	7	186	71.0	72.6	184	85.1	86.3	182	81.4	84.2	184	84.1	85.8	196	43.1	52.3	195	52.5	57.0	206	34.0	37.4	193	45.6	50.8	213	60.5	67.0
	6	190	67.4	65.5	187	83.0	81.3	186	80.3	78.4	184	83.8	80.7	198	46.6	45.0	202	50.4	47.0	211	33.7	32.2	209	41.3	35.5	221	46.7	61.4
	5	194	61.1	63.3	189	72.4	75.0	188	68.8	70.8	187	70.3	74.2	206	35.9	38.5	206	33.1	34.8	212	27.5	28.6	216	18.5	22.5	223	51.5	55.3
	4	195	61.1	60.4	191	67.4	67.5	192	61.9	62.0	191	66.8	66.8	212	32.1	28.0	213	29.9	26.5	209	24.9	26.7	223	16.8	13.9	225	43.8	48.0
	3	196	55.4	56.8	195	53.9	63.3	194	55.4	53.8	195	55.7	60.0	217	15.4	19.7	217	14.8	18.7	217	18.7	21.2	226	6.6	7.9	226	37.9	39.8
	2	199	55.0	52.0	198	58.5	56.4	198	54.4	53.4	199	54.7	50.8	223	14.6	11.5	223	14.3	10.8	223	17.4	13.0	230	5.3	4.5	228	32.1	31.0
	1	203	44.7	44.0	204	46.6	45.8	201	46.0	44.4	204	39.1	38.6	227	5.9	6.0	227	5.1	5.5	228	6.0	6.0	232	2.4	2.7	228	21.1	19.7
Reboiler		205	29.0	29.0	207	30.3	30.3	204	31.2	31.0	209	25.4	25.4	218	2.4	2.5	218	2.2	2.2	224	2.4	2.4	226	1.8	1.8	219	6.3	6.5
Condenser - H_2O in		90			91			89			88			85			86			88			86			86		
- H_2O out		112			115			121			134			127			127			121			118			134		
- Prod. out		111			112			115			134			132			132			125			126			145		
Feed		89	53.7		86	–		83	52.2		82	51.1		84	57.8		83	57.1		85	54.8		–			–		
Control Air Press. psig.		6.8			7.1			5.5			10.6			23.5			23.5			22.25			22.5			21.75		
Steam Press. - psig.		5.5			5.5			4			9			21			23.5			20.5			–			–		
Reflux	gpm.	0.09			0.09			0.07			0.07			0.08			0.08			0.070			0.218			0.185		
Product	gpm	0.062			0.057			0.052			0.063			0.078			0.078			0.084			0.023			0.033		
Feed	gpm	0.075 #/min.			0.85 #/min.			0.108			0.49 #/min.			0.150			0.150			0.110			0.023			0.093		
ΔP across col.	"Hg.	0.9			0.8			0.7			0.8			0.9			1.1			1.2			–			–		
Feed Plate		6			4			4			4			6			8			4			5			5		

SAMPLE CALCULATION

Run 29: Feed Introduced on Plate 4 (Above Still)

$V = R + P = 0.070 + 0.063 = 0.133$

x_V = 99.4% benzene $t_R = 114^{\circ}F.$

$R = (0.070)(8.33)(0.875) / (78.2) = 0.00653$ mols/min.

$V = (0.0653)(0.133) / (0.070) = 0.01240$ mols/min.

Enthalpies of vapor and reflux:

H at 114° = (0.99)(2706) + (0.01)(3095) = 2710 Btu/mol
H at B.P. = = 4930 Btu/mol Δ = 2220
H of vapor = = 18150 Btu/mol Δ = 13220

Vapor condensed by cold reflux = (0.0065)(2220) / 13220 =

0.0011 mols/min.

Effect of column heat loss on enriching line:

Heat loss above feed plate = 45 Btu/min.

Average Composition = 88.5% benzene

H of vapor = 18,610 Btu/mol.
H of liquid = 5,160 Btu/mol. Δ = 13,450 Btu/mol.

Mols condensed per minute in 8-plates above feed =

45 / 13,450 = 0.0034

Mols condensed per minute in a 4 plate section = 0.0017 mols/min.

	Top Plate	Plate 8	Above Feed
L	0.0065	0.0076	0.0093
	11	17	17
	0.0076	0.0093	0.0110
V	0.0124	0.0135	0.0152
	11	17	17
	0.0135	0.0152	0.0169
L/V	0.562	0.612	0.651
$L/V_{avg.}$	0.585	0.630	

See Table 10 for original data

TABLE II

CALCULATIONS SUMMARY — PARTIAL REFLUX RUNS

RUN No.	Overhead Prod. Mol % O	Bottoms Mol % O	Feed. Pl. Liquid Mol % O	Feed Mol % O	Feed Plate	Avg. Plate Comp. Mol % O	Vapor Velocity at top of Col. ft per sec.	V to Condr. mols/min. x10⁴	R to Column mols/min x10⁴	P mols/min x10⁴	V Cond. by R mols/min x10⁴	V Cond. by Heat Loss above Feed mols/min. x10⁴	(L/V) at top Plate	(L/V) Intermed. in Enrich. Section	(L/V) Above Feed Plate	(L/V) Avg. Top of Enrich. Sec.	(L/V) Avg. Bot. of Enrich Sec.	Theoretical Plates	Overall Efficiency	Correction to Eff. for Vapor Velocity	Xavg − 65	Correction to Eff. for Composition	Total Correction; Vap. Vel & Comp.	Efficiency Corrd to V = 1'/sec. and Xavg = 65% O	Theoret. Location of Feed Plate Frac. from Col. Top	Actual Location of Feed Plate Frac. fr. Col. Top.	Ratio Feed Plate Location to Theoretical
15	95.0	17.5	78.7	57.1	10	67.2	0.455	[illegible]	92	50	22	11	0.695	-	0.714	0.705	-	5.70-1	39.2%	O	2.2	1.3	1.3	40.5	0.565	0.231	0.409
16	97.7	15.0	89.4	63.9	10	73.7	0.922	267	228	39	50	11	0.876	-	0.880	0.88	-	6-1	41.6	O	8.7	5.2	5.2	46.8	0.550	0.231	0.419
17	98.8	13.5	93.7	69.2	10	76.2	1.505	454	404	50	76	11	0.906	-	0.908	0.91	-	6.71-1	47.6	O	11.2	6.7	6.7	54.3	0.535	0.231	0.432
18	95.6	16.5	81.0	66.4	10	70.6	0.496	141	93	48	15	11	0.692	-	0.712	0.70	-	5.69-1	39.1	O	5.6	3.4	3.4	42.5	0.459	0.231	0.503
19	95.0	15.0	77.0	61.9	10	66.3	0.432	124	74	50	17	11	0.645	-	0.671	0.66	-	6.36-1	44.7	O	1.3	0.8	0.8	45.5	0.647	0.231	0.357
20	94.2	13.5	75.1	63.5	10	63.8	0.432	124	74	50	17	11	0.645	-	0.671	0.66	-	6.25-1	43.7	-1.3	-1.2	-0.7	-2.0	41.7	0.460	0.231	0.502
21	94.6	4.0	53.5	67.4	6	51.3	0.504	143	83	50	13	25	0.615	0.645	0.678	0.63	0.66	10.6-1	80.0	O	-13.7	-8.3	-8.3	71.7	0.313	0.538	1.722
23	98.8	33.5	69.5	57.1	6	72.9	0.438	125	75	50	15	26	0.643	0.673	0.698	0.66	0.68	7.69-1	55.7	O	7.9	4.8	4.8	60.5	0.797	0.538	0.677
25	96.2	24.5	66.8	57.4	8	68.4	0.496	141	84	57	16	18	0.662	0.697	0.697	0.68	-	6.31-1	44.3	O	3.4	2.1	2.1	46.4	0.657	0.385	0.586
26	97.7	29.0	65.5	53.7	6	71.6	0.496	141	84	57	16	26	0.637	0.663	0.688	0.65	0.675	7.03-1	50.2	O	6.6	4.0	4.0	54.2	0.756	0.538	0.713
27	98.8	30.3	67.5	-	4	78.9	0.480	137	84	53	16	34	0.653	0.688	0.716	0.67	0.70	7.69-1	55.7	O	13.9	8.4	8.4	64.1	0.663	0.692	1.042
28	99.4	31.0	62.0	52.2	4	76.5	0.395	114	65	49	12	34	0.611	0.657	0.693	0.635	0.675	9.84-1	73.7	O	11.5	6.9	6.9	80.6	0.837	0.692	0.827
29	99.4	25.4	66.8	51.1	4	77.4	0.435	124	65	59	11	34	0.562	0.612	0.651	0.585	0.630	10.4-1	77.0	-1.5	12.4	7.5	6.0	83.0	0.635	0.692	1.089
32	92.3	2.5	45.0	57.8	6	48.1	0.516	145	74	71	12	25	0.547	0.579	0.610	0.56	0.60	15.8-1	132.7	O	-16.9	-10.4	-10.4	122.3	0.318	0.538	1.695
33	92.7	2.2	65.0	57.1	8	49.2	0.516	145	74	71	12	18	0.547	-	0.606	0.575	-	10-1	75.0	O	-15.8	-9.5	-9.5	65.5	0.337	0.385	1.142
34	84.2	2.4	26.7	51.8	4	51.8	0.504	140	64	76	12	32	0.500	0.547	0.587	0.525	0.567	Indet.	Indet.	O	-13.2	-8.0	-8.0	Indet.			
35	91.9	1.8	-	91.9	5	44.8	0.788	220	199	21	35	42	0.918	-	0.929	0.925	-	Indet.	Indet.	O							
36	83.5	6.5	-	83.5	5	59.4	0.713	198	168	30	27	42	0.865	-	0.888	0.875	-	Indet.	Indet.	O							

TABLE 12
MURPHREE EFFICIENCY CALCULATIONS

RUN No.	PLATE No.	x	y	y^*	$y_n - y_{n-1}$	$y^*_n - y_{n-1}$	η
5	8	96.7	98.7	98.8	2.0	2.1	95.3
	7	94.1	96.7	98.0	2.6	4.0	65.0
	6	90.0	94.1	96.3	4.1	6.3	65.2
	5	84.0	90.0	93.5	6.0	9.0	66.7
	4	75.8	84.0	88.8	8.2	13.0	63.1
	3	65.5	75.8	82.7	10.3	17.2	59.9
	2	52.5	65.5	73.4	13.0	20.9	62.2
	1	36.9	52.5	59.0	15.6	22.1	70.6
	W	19.5	36.9				
6	11	95.8	98.2	98.3	2.4	2.5	96.0
	10	92.8	95.2	97.1	3.0	4.3	69.8
	9	88.8	92.8	95.2	4.0	6.4	62.5
	8	84.3	88.8	93.2	4.5	8.9	50.6
	7	79.1	84.3	90.7	5.2	11.6	44.8
	6	73.7	79.1	87.9	5.4	14.2	38.1
	5	67.0	73.7	84.0	6.7	17.0	39.4
	4	59.7	67.0	78.8	7.3	19.1	38.2
	3	51.1	59.7	71.9	8.6	20.8	41.3
	2	41.6	51.1	63.4	9.5	21.8	43.6
	1	30.0	41.6	50.3	11.6	20.3	57.2
	W	9.0	30.0				
7	8	93.0	96.8	97.2	3.8	4.2	90.5
	7	88.5	93.0	95.1	4.5	6.6	68.2
	6	82.8	88.5	92.6	5.7	9.8	58.3
	5	75.0	82.8	88.6	7.8	13.6	57.3
	4	67.7	75.0	84.4	7.3	16.7	43.7
	3	60.0	67.7	79.0	7.7	19.0	40.6
	2	51.5	60.0	73.0	8.5	21.5	39.5
	1	39.5	51.5	61.2	12.0	21.7	55.3
	W	14.3	39.5				

RUN No	PL. No.	x	y	y^*	$y_n - y_{n-1}$	$y^*_n - y_{n-1}$	η
10	8	96.4	98.5	97.2	2.1	2.4	87.5
	7	93.7	96.4	95.1	2.7	4.1	65.8
	6	90.2	93.7	92.6	3.5	5.8	60.4
	5	85.0	90.2	88.6	5.2	9.0	57.8
	4	76.5	85.0	84.4	8.5	12.7	65.9
	3	65.2	76.5	79.0	11.3	17.3	65.3
	2	50.8	65.2	73.0	14.4	21.6	66.7
	1	34.0	50.8	61.2	16.8	22.0	79.8
	W	15.0	34.0				
21	12	87.0	94.5	94.8	4.9	5.2	94.3
	11	80.7	89.6	92.6	3.9	6.9	56.5
	10	75.0	85.7	88.7	3.7	6.7	55.2
	9	69.4	82.0	85.2	3.7	6.9	53.7
	8	64.1	78.3	81.8	3.3	6.8	48.5
	7	58.8	75.0	78.2	4.0	7.2	55.5
	6	53.5	71.0	74.2	3.4	6.6	51.5
	5	43.5	67.6	65.8	13.1	11.3	118.0
	4	33.6	54.5	55.5	12.5	13.5	92.7
	3	24.3	42.0	43.5	10.0	13.5	74.2
	2	16.0	30.0	31.2	11.0	12.2	90.2
	1	9.3	19.0	19.4	8.5	8.9	95.5
	W	4.0	10.5	8.5			
24	7	96.7	98.2	99.0	1.5	2.3	65.2
	6	94.8	96.7	98.0	1.7	3.0	56.7
	5	91.9	94.8	97.0	3.2	5.2	61.5
	4	87.3	91.9	95.0	4.6	7.8	59.0
	3	81.0	87.3	91.8	6.2	10.8	57.4
	2	71.7	81.0	86.7	9.5	15.2	62.5
	1	56.3	71.7	76.3	15.2	20.0	75.9
	W	36.0	56.3	58.2			

RUN No.	PL. No.	x	y	y^*	$y_n - y_{n-1}$	$y^*_n - y_{n-1}$	η
25	12	92.6	96.2	97.3	2.4	3.5	68.6
	11	88.8	93.8	94.7	2.8	3.7	75.7
	10	84.5	91.0	93.5	3.0	5.5	54.6
	9	79.1	88.0	90.8	3.8	6.6	57.6
	8	70.3	84.2	85.7	6.0	7.5	80.0
	7	68.6	78.2	84.7	1.2	7.7	15.6
	6	66.8	77.0	83.5	1.0	7.5	13.3
	5	64.6	76.0	82.0	3.0	9.0	33.3
	4	61.6	73.0	80.2	3.5	10.7	32.7
	3	52.8	69.5	73.7	10.7	14.9	71.8
	2	51.1	58.8	72.2	2.0	15.4	13.0
	1	40.5	56.8	62.6	13.2	19.0	69.4
	W	24.5	43.6				
35	12	89.5	91.9	96.0	2.2	6.3	34.9
	11	86.7	89.7	94.7	2.7	7.7	35.1
	10	82.2	87.0	92.5	3.8	9.3	40.8
	9	75.6	83.2	88.8	6.4	12.0	53.3
	8	65.0	76.8	82.5	9.8	15.5	63.2
	7	50.8	67.0	72.0	13.5	18.5	72.9
	6	35.5	53.5	57.5	14.3	18.3	78.2
	5	22.5	39.2	41.0	11.7	13.5	86.7
	4	13.9	27.5	27.5	8.0	8.0	100.0
	3	7.9	19.5	16.5			

TABLE 13
RECALCULATION OF MURPHREE EFFICIENCIES TO LIQUID FILM BASIS

Run No	Pl. No.	X	η	k	m	k/m	η
10	8	96.4	87.5	2.085	0.40	5.21	99.45
	7	93.7	65.8	1.070	0.415	2.575	92.32
	6	90.2	60.4	0.930	0.435	2.135	88.2
	5	85.0	57.8	0.865	0.465	1.860	84.4
	4	76.5	65.9	1.075	0.560	1.92	85.3
	3	65.2	65.3	1.060	0.645	1.645	80.7
	2	50.8	66.7	1.100	0.830	1.325	73.5
	1	34.2	79.8	1.605	1.12	1.432	76.0
21	12	87.0	94.3	2.870	0.460	6.24	99.8
	11	80.7	56.5	0.835	0.505	1.653	80.8
	10	75.0	55.2	0.805	0.550	1.463	76.8
	9	69.4	53.7	0.770	0.600	1.282	72.2
	8	64.1	48.5	0.652	0.660	0.987	62.7
	7	58.8	55.5	0.812	0.715	1.135	67.8
	6	53.5	51.5	0.723	0.790	0.915	59.8
	5	43.5	118.0	9	0.945	0.953	61.3
	4	33.6	92.7	2.625	1.135	2.31	90.2
	3	24.3	74.2	1.353	1.350	1.00	63.2
	2	16.0	90.2	2.315	1.630	1.42	75.7
	1	9.3	95.5	3.100	1.90	1.63	80.3
35	12	89.5	34.9	0.43	0.440	0.978	62.3
	11	86.7	35.1	0.433	0.465	0.932	60.6
	10	82.2	40.8	0.525	0.495	1.16	68.6
	9	75.6	53.3	0.762	0.54	1.41	78.0
	8	65.0	63.2	1.00	0.65	1.54	78.5
	7	50.8	72.9	1.308	0.83	1.58	79.3
	6	35.5	78.2	1.530	1.10	1.39	75.0
	5	22.5	86.7	2.02	1.41	1.43	76.0
	4	13.9	100	9	1.71	5.25	99.5

TABLE 14

THERMOCOUPLE INSTALLATION

Switch Number	Location
1	Steam calorimeter inlet
2	Cooler-water out
3	Product in
4	Reboiler
5	Feed
6	Column-plate 4
7	-plate 3
8	-plate 2
9	-plate 1
10	Cooler-water in
11	Steam calorimeter out
12	Cooler-product out
13	Column-plate 10
14	-plate 9
15	-plate 8
16	-plate 7
17	-plate 6
18	-plate 5
19	Condenser-water in
20	-water out
21	-condensate out
22	Column-vapor out
23	-plate 12
24	-plate 11

VI

NOMENCLATURE AND SYMBOLS

Symbol	Term	Units
A	Interfacial Area	
C_p	Specific Heat at Constant Pressure	Btu./lb.
D	Distillate Rate	Lb.-mol/min.
d	Differential Operator	
e	Base of Natural Logarithms	
F	Feed Rate	Lb.-mol/min.
f_V, f_L	Liquid and Vapor film Coefficients	
H	Enthalpy	Btu./lb.-mol
h	Enthalpy	Btu./lb.
k	Constant or Exponent	
L	Liquid Rate	Lb.-mol/min.
M	Molecular Weight	
m	Slope of x-y Equilibrium Curve	
n	Mol Fraction	
n, n+1	Subscripts referring to n and n+1 plates	
P	Pressure or vapor Pressure	Various
T	Temperature, absolute	^{o}R or ^{o}K
t	Temperature, usual	^{o}F or ^{o}C
V	Vapor Rate	Lb.-mol/min.
W	Bottoms Rate	Lb.-mol/min.
x, X	Mol Fraction Benzene in Liquid	
y, Y	Mol fraction Benzene in Vapor	

Symbol	Term	Units
y^*	Mol Fraction Benzene in Vapor at Equilibrium	
η , η_{OA}	Efficiency; overall plate efficiency	
$\eta_{M,V}$, $\eta_{M,L}$	Murphree Efficiency, Vapor or Liquid Film Controlling	
η_y, η_x	Murphree Efficiency Calculated on Vapor and Liquid Bases	
θ	Time, time of contact	
λ	Latent Heat of Vaporization	Btu./lb.
μ	Gas Compressibility Factor	

VI

LITERATURE CITED

1. Baker, E. M. and Lindsay, R. A., Ind. Eng. Chem. 35, 418 (1943)
2. Barker, J. T., Z. Physik. Chem. 71, 235 (1910)
3. Baud, E., Bull. soc. chim. 17, 329 (1915)
4. Bowden, R. H., M.S. in Ch. E. Thesis, Univ. of Texas (1945)
5. Brown, G. G. and Lockhart, F. J., Trans. Am. Inst. Chem. Engrs. 39, 63 (1943)
6. Brown, G. G., Ibid. 32, 321 (1936)
7. Brown, G. G. and Martin, H. Z., Ibid. 35, 679 (1939)
8. Carey, J. S., Griswold, J., Lewis, W. K. and McAdams, W. H., Ibid. 30, 504 (1934)
9. Colburn, A. P., Ind. Eng. Chem. 33, 459 (1941)
10. Colburn, A. P., Trans. Am. Inst. Chem. Engrs. 35, 211 (1939)
11. Drickamer, H. G. and Bradford, J. R., Ibid. 39, 319 (1943)
12. Egloff, G. and Lowry, Jr., C. D., Ind. Eng. Chem. 21, 920 (1929)
13. Fiock, E. F., Ginnings, D. C. and Holton, W. B., Bur. Stds. J. Res. 6, 881 (1921)
14. Gay, L., Chimie & industrie 6, 567 (1921)
15. Gerster, J. A., Koffolt, J. H. and Withrow, J. R., Trans. Am. Inst. Chem. Engrs. 39, 37 (1943)
16. Griswold, J., Sc. D. Thesis, M. I. T. (1931); Trans. Am. Inst. Chem. Engrs. 35, 588 (1939)
17. Griswold, J., Andres, D. and Klein, V. A., Trans. Am. Inst. Chem. Engrs. 39, 223 (1943); Refiner 22, 171 (1943)
18. Hausbrand, E., "Die Wirkungsweise der Rektifizier- und Destillierapparate" Verlag von Julius Springer, Berlin (1921)
19. Holbrook, G. E. and Baker, E. M., Trans. Am. Inst. Chem. Engrs. 30, 520 (1934)

20. Kelley, K. K., J. Am. Chem. Soc. 51, 2738 (1929)

21. Keyes, D. B. and Byman, L., Univ. Illinois Bull. Eng. Expt. Sta., Bull Ser. 38, No. 328 (1941); Chem. Met. Eng. 48, No. 7, 85 (1941)

22. Kirschbaum, E., "Destillier- und Rektifiziertechnik" Verlag von Julius Springer, Berlin (1940). Reprinted by Edwards Bros., Ann Arbor, Mich. (1943)

23. Ibid., p. 211

24. Kirschbaum, E. and Andrews, C. A., J. Inst. Petroleum Tech. 22, 803 (1936)

25. Krase, N. W. and Goodman, J. B., Ind. Eng. Chem. 22, 13 (1930)

26. Langdon, W. M. and Keyes, D. B., Ibid. 35, 464 (1943)

27. Lewis, W. K., Ibid. 14, 492 (1922)

28. Lewis, Jr., W. K., Ibid. 28, 399 (1936)

29. Mathews, J. H., J. Am. Chem. Soc. 48, 562 (1926)

30. McCabe, W. L. and Thiele, E. W., Ind. Eng. Chem. 17, 605 (1925)

31. Montgomery, J. B. and DeVries, T., J. Am. Chem. Soc. 64, 2372 (1942)

32. Murphree, E. V., Ind. Eng. Chem. 17, 747 (1925)

33. National Bureau of Standards, "Selected Values of Properties of Hydrocarbons" Table 5, A.P.I. Research Project 44, Am. Pet. Inst., New York (1943)

34. National Research Council, "International Critical Tables" 3, 223, McGraw-Hill Book Co., New York (1928)

35. Ibid. 5, 12

36. Ibid. 5, 128

37. Ibid. 5, 157

38. Ibid. 7, 218

39. Othmer, D. B., Chem. Met. Eng. 49, No. 5, 84 (1942)

40. Othmer, D. F., Ind. Eng. Chem. 32, 841 (1940)

41. Peavy, C. C. and Baker, E. M., Trans. Am. Inst. Chem. Engrs. 33, 315 (1937)

42. Peirce, B. O., "A Short Table of Integrals," 3rd Ed., Ginn and Co., Boston (1929), p. 127

43. Peters, Jr., W. A., Ind. Eng. Chem. 14, 476 (1922)

44. Randall, M. and Longtin, B., Ibid. 31, 908 (1939)

45. Rhodes, F. H. and Slachman, P. G., Ibid. 29, 51 (1937)

46. Robinson, C. S., Ibid. 14, 480 (1942)

47. Robinson, C. S., and Gilliland, E. R., "The Elements of Fractional Distillation," 3rd Ed., McGraw-Hill Book Co., New York (1939)

48. Ibid., p. 91

49. Rosanoff, M. A., Bacon, C. W. and Schulze, J. F. W., J. Am. Chem. Soc. 36, 1993 (1914)

50. Sherwood, T. K. and Jenny, F. J., Ind. Eng. Chem. 27, 265 (1935)

51. Singer, Jr., S. C., Wilson, R. R. and Brown, G. G., Ibid. 28, 824 (1936)

52. Smith, R. H. and Andrews, D. H., J. Am. Chem. Soc. 55, 3644 (1931)

53. Sorel, E. "La Rectification de l'Alcool" --------, Paris (1893)

54. Stull, D. R., Ind. Eng. Chem. 35, 1303 (1943)

55. Underwood, A. J. V., Trans. Inst. Chem. Engrs. (London) 10, 112 (1932)

56. Watson, K. M. and Fallon, Nat. Petr. News 36, R. 372 (June 7, 1944)

57. Williams, J. W. and Daniels, F., J. Am. Chem. Soc. 46, 903 (1924)

Typed by Evelyn C. Bennett

Typed by Evelyn C. Painter

The vita has been removed from the digitized version of this document.